

Appendix C

SOPs

LABORATORY TESTING OF VENTILATION FAN PERFORMANCE
Standard Operating Procedure (SOP) A1

LABORATORY TESTING OF VENTILATION FAN PERFORMANCE

Standard Operating Procedure (SOP) A1

Prepared by

Steve E. Ford (Univ. of Illinois Urbana-Champaign) and Bill W. Bogan

Reviewed by

**Yuanhui Zhang (Univ. of Illinois at Urbana-Champaign), Steve J. Hoff (Iowa State Univ.),
and Albert J. Heber**

Effective Date: November 6, 2006

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability.....	3
2. Summary of Method.....	3
3. Definitions.....	3
4. Health and Safety.....	4
5. Cautions.....	4
6. Interferences.....	4
7. Personnel Qualifications.....	4
8. Equipment and Supplies.....	4
9. Procedures.....	5
10. Data and Records Management.....	6
11. Quality Control and Quality Assurance.....	6
12. References.....	7
Appendix A. Fan test datasheet.....	9

1. Scope and Applicability

- 1.1. This SOP describes performance testing of ventilation fans that are used on mechanically-ventilated barns and livestock buildings, including manure sheds.
- 1.2. Fan testing determines performance in terms of airflow rate, pressure, speed of rotation, power and ventilation efficiency.
- 1.3. The methodology described in this SOP is suitable for the testing of wall-mounted, constant- or variable-speed ventilation fans, including manure storage pit fans. It does not apply to the testing of air circulating fans.
- 1.4. Testing is performed in accordance with two industry standards:
 - 1.4.1. American Society of Agricultural and Biological Engineers (ASABE) Standard S565 (“Agricultural Ventilation Constant Speed Fan Test Standard”)
 - 1.4.2. Air Movement and Control Association (AMCA) Standard 210-99 (“Laboratory Methods of Testing Fans for Rating”) – Equivalent to American Society of Heating, Refrigeration and Air-conditioning Engineers (ASHRAE) Standard 51-1999
- 1.5. Testing of the fans is conducted using a stationary laboratory fan test facility located in the Bioenvironmental and Structural Systems (BESS) Laboratory, Agricultural Engineering Sciences Building, University of Illinois at Urbana-Champaign.

2. Summary of Method

An airflow test chamber was designed and constructed according to AMCA 210-99/ASHRAE 51-1999 standard format (Fig. 15 of AMCA 210-99, included as Fig. 1 at the end of this SOP). The airflow test chamber measures airflow in the range of 100 - 28,000 ft³/min (accuracy $\pm 2\%$). The fan being tested is mounted in the chamber, and the volume of air handled is measured at up to six distinct static pressures by varying the air supplied by the chamber’s variable supply system. This is done by measuring the pressure differential across the nozzle in the center of the chamber, which is proportional to the volume of air moved by the fan. Electrical measurements (voltage and current) are made with a Valhalla Scientific 2100 power analyzer (for single-phase testing) or a Valhalla Scientific 2300 power analyzer (for three-phase testing). The rotational speed of the fan impeller is measured with either a digital stroboscope (Nidec-Shimpo Model DT-301) or a fiber-optic photoelectric sensor (Banner Model SM312FMHS). Wet and dry bulb temperature and barometric pressure are measured so that air density may be calculated. Fan ventilation efficiency ratio (VER) is calculated in units of ft³/min-W.

3. Definitions

- | | |
|-------------------------|--|
| 3.1. BESS | Bioenvironmental and Structural Systems |
| 3.2. Manure pit fan | A ventilation fan used to remove harmful gases from a manure pit, and which is designed to resist corrosion caused by these gases. |
| 3.3. PAAQL | Purdue Agricultural Air Quality Laboratory |
| 3.4. Variable-speed fan | A fan whose speed can be modulated, usually based on temperature. |

- 3.5. Ventilation efficiency ratio (VER) Airflow volume per unit of power consumed by the operation of the fan. Units used for VER in this SOP are ft^3/W .
- 3.6. Ventilation fan An un-ducted, axial-flow fan system used for air exchange (as opposed to air circulation) purposes. Generally consists of an impeller, housing, motor, shutter or damper, and guard.

4. Health and Safety

- 4.1. Fans should not be plugged in until the test and warm-up period is started.
- 4.2. Wear safety glasses while the fan is operating.

5. Cautions

- 5.1. Since the air chamber is under negative pressure, any leaks between the flow nozzles and the fan will cause a negative bias in fan airflow results. Make sure that there are no leaks in the chamber, or in the mounting seal. Ensure all joints around the fan assembly and the chamber are sufficiently tight. Test for leaks in the chamber by sealing up the fan opening (no fan installed) and pressurizing the chamber while introducing smoke into the chamber. Leaks are readily identified by visualizing the smoke.
- 5.2. Fans should be mounted for testing in the same configuration as they are installed or deployed in the field. All protective screens or guards, hoods or other wind-deflecting attachments, or discharge cones should be in-place during testing. Failure to do so may produce data which are not truly applicable to the field performance of the fan.

6. Interferences

- 6.1. In order to avoid inaccurate airflow data due to blockage, the exhaust area of the fan being tested should be kept clear (to a distance of at least three times the fan diameter).

7. Personnel Qualifications

- 7.1. Personnel should be trained in the operation of all instruments listed in Sections 8.1 through 8.6.1. The training will be provided by PAAQL and UIUC BESS Laboratory personnel.
- 7.2. Personnel must read and understand all applicable instrument manuals and this SOP before operating any of the equipment. These documents will be kept at the UIUC BESS.

8. Equipment and Supplies

- 8.1. Airflow test chamber as specified in AMCA 210/99 (See Fig. 1)
- 8.2. Variable voltage transformer capable of delivering 230 V with a precision of ± 0.5 V
- 8.3. Manometer (Model 40HE35WM, Meriam Process Technologies, Cleveland, OH)

- 8.4. Micro-manometer (Model 1430, Dwyer Instruments, Michigan City, IN)
- 8.5. Mechanically aspirated psychrometer (Model Series 37210, Oakton Instruments, Vernon Hills, IL)
- 8.6. Barometer (Model 469, Princo Instruments, Southampton, PA)
- 8.7. Power analyzer (Model 2100 or 2300, Valhalla Scientific, San Diego, CA)
- 8.8. Fan impeller rotational speed monitor (two options)
 - 8.8.1. Digital stroboscope (Model DT-301, Nidec-Shimpo America Corp, Itasca, IL)
 - 8.8.2. Fiber-optic photoelectric sensor (Model SM312FMHS, Banner Engineering, Minneapolis, MN)

9. Procedures

- 9.1. Fan Performance Test
 - 9.1.1. Instrument checks (To be performed prior to each series of tests)
 - 9.1.1.1. Manometer
 - 9.1.1.1.1. Check fluid level according to instrument manual.
 - 9.1.1.1.2. With high and low pressure manifolds shunted, zero the instrument by adjusting the level until the micromanometer reads zero.
 - 9.1.1.2. Psychrometer
 - 9.1.1.2.1. Turn instrument on and observe the wet bulb temperature reading.
 - 9.1.1.2.2. Replace batteries if it takes more than 2 min for the wet bulb temperature to stabilize.
 - 9.1.1.3. Barometer
 - 9.1.1.3.1. Check the white zero pointer in the cistern and adjust to zero according to the instrument manual.
 - 9.1.2. Test procedure
 - 9.1.2.1. Mount the fan in the test chamber, with the fan inlet facing the main chamber, as shown in Fig. 1. All joints between the fan assembly and the chamber itself must be sufficiently tight that air leakage does not occur during the test. Leaks can be detected with a tufted wand while the chamber is pressurized with the chamber air supply.
 - 9.1.2.2. Check and zero the manometers before the fan is turned on.
 - 9.1.2.3. Turn the fan motor on and ensure that the input voltage is 230 V. Allow the fan motor to operate 20 min before proceeding with test.
 - 9.1.2.4. Measure and record wet-bulb and dry-bulb air temperature of the lab with the psychrometer.
 - 9.1.2.5. Measure and record the barometric pressure.
 - 9.1.2.6. Turn on the chamber air supply fan.
 - 9.1.2.7. Set the fan static pressure at free air (0" static pressure), which is to equalize the pressure in the chamber immediately upstream of the test fan with the pressure in the lab area surrounding the chamber. This is conducted by adjusting the operating static pressure point of the test fan by adjusting the supply fan blade pitch and by-pass door. Large changes in static pressure should be performed by blade pitch adjustment. Adjust the by-pass door for small changes.

- 9.1.2.8. The pressure drop (ΔP) across the measurement nozzles should remain above 0.5 inches of water gauge (iwg). The number of nozzles open must be lowered if the measurement falls below 0.5 iwg. If only one nozzle is open and the pressure differential drops below 0.5 iwg, select a smaller nozzle size.
- 9.1.2.9. Record the number of flow nozzles open, nozzle diameter (in.), pressure drop across the flow measurement nozzles (in. of water), fan speed (rpm), voltage (V), current (A), and power (W). A sample data sheet is included in Appendix A of this SOP.
 - 9.1.2.9.1. Take readings when the system is at equilibrium. Equilibrium can be recognized by observing the stable pressure and other variable readings, or by taking trail measurements of the various variables until constant values are obtained. Equilibrium is usually reached within one to five minutes when the system is warmed-up. Note any test conditions (static pressures) under which equilibrium cannot be reached.
- 9.1.2.10. Increase the fan static pressure by 0.05 iwg, and repeat steps 9.1.2.7 through 9.1.2.9. The fan should be tested for at least 5 points, from 0.0 iwg up to 0.25 iwg static pressure, or airflow cut-off, whichever comes first. The former of these would result in a total of six static pressure points being tested (0, 0.05 iwg, 0.10 iwg, 0.15 iwg, 0.20 iwg, and 0.25 iwg).

10. Data and Records Management

- 10.1. Appendix A of this SOP contains a sample data sheet to be used for recording the data collected in these tests.
- 10.2. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
- 10.3. Manage all data according to SOP B5.
- 10.4. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes.

11. Quality Control and Quality Assurance

- 11.1. Equipment manuals & certification
 - 11.1.1. Master copies of instruction manuals and calibration certificates (when applicable) for all instruments described herein are kept in a specific file drawer, and made available to the users during training and measurement.
 - 11.1.2. The fan test data will be kept at the UIUC BESS and delivered to PAAQL. The PAAQL will distribute the data to the trailer at the respective farm.
- 11.2. Equipment performance checks and calibration
 - 11.2.1. Manometer
 - 11.2.1.1. Calibrate with the micro-manometer annually according to manufacturer's procedures. Perform the following steps on the micro-manometer before using:
 - 11.2.1.1.1. Check fluid level.
 - 11.2.1.1.2. Remove needle tip. Clean and reinstall.

11.2.1.1.3. Check battery and replace (AA) if necessary.

11.2.2. Psychrometer

11.2.2.1. Calibrate thermometers annually. Calibrate four points between 32°F and 100°F in a water bath with either a NIST-traceable thermometer or a block calibrator (Model DB-35L, Techne Incorporated, Burlington, NJ).

11.2.3. Power analyzer

11.2.3.1. Cross check performance of the Valhalla Scientific 2100 or 2300 power analyzer with a power transducer (Model EW5–20B, Ohio Semitronics, Hilliard, OH). Voltage and current should be calibrated against a multimeter (Model 75 or Model 77, Fluke Corp., Everett, WA). Both of these checks should be done annually.

12. References

- 12.1. AMCA Standard 210-99. 1999. Laboratory Methods of Testing Fans for Rating. Air Movement and Control Association, Inc., Arlington Heights, IL.
- 12.2. ASABE Standard S565. 2005. Agricultural Ventilation Constant Speed Fan Test Standard. American Society of Agricultural and Biological Engineers, St. Joseph, MI.
- 12.3. Barometer Manual. Operations Manual for Model 469 Barometer. Princo Instruments, Southampton, PA.
- 12.4. Manometer Manual. Operations Manual for Model 40HE35WM Manometer. Meriam Process Technologies, Cleveland, OH.
- 12.5. Micro-Manometer Manual. Operations Manual for Model Model 1430 Micro-Manometer. Dwyer Instruments, Michigan City, IN
- 12.6. Power Analyzer Manual. Operations Manual for Model 2100 or 2300. Valhalla Scientific, San Diego, CA.
- 12.7. Psychrometer Manual. Operations Manual for Model Series 37210 Mechanically Aspirated Psychrometer. Oakton Instruments, Vernon Hills, IL
- 12.8. Stroboscope Manual. Operations Manual for Model DT-301 Digital Stroboscope. Nidec-Shimpo America Corp, Itasca, IL
- 12.9. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.

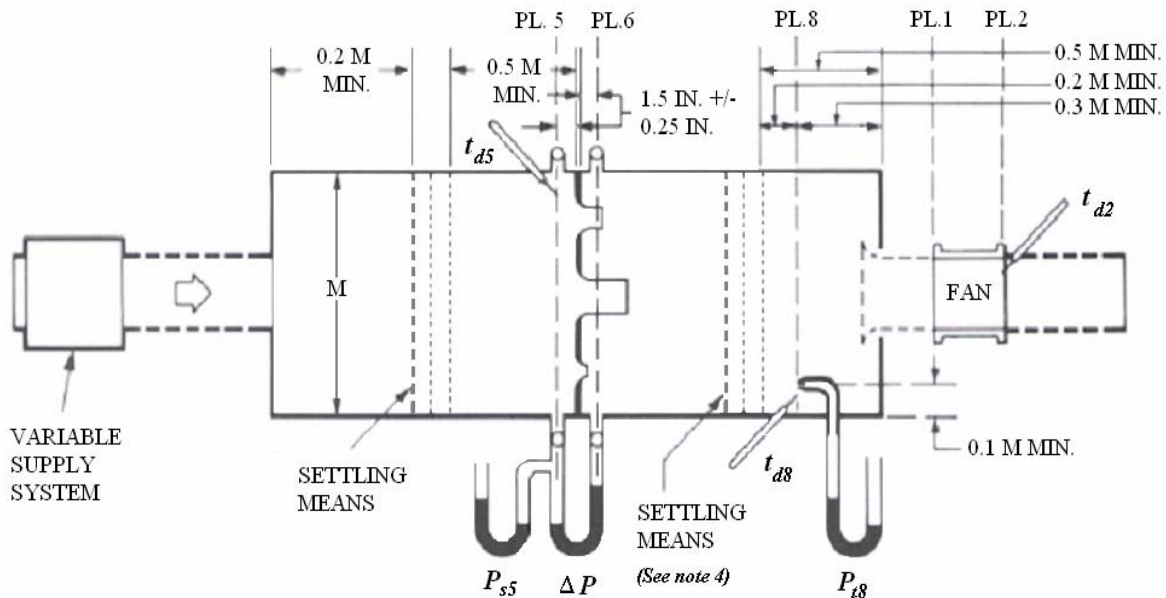


Figure 1. Configuration of the fan testing chamber, as adapted from AMCA210-99 Figure 15. M = Main chamber dimension (ft); PL. = Plane; P_s = Static pressure; t_d = Dry-bulb temperature; ΔP = Pressure differential; Settling means = mesh screen to create uniform flow.

Notes:

1. Dotted lines on fan inlet indicate an inlet bell (bell-mouth inlet that is fabricated of steel or alloy with flange) and one equivalent duct diameter which may be used for inlet duct simulation. The duct friction shall not be considered.
2. Dotted lines on fan outlet indicate a uniform duct 2 to 3 equivalent diameters long, and of an area within $\pm 0.5\%$ of the fan outlet area and a shape to fit the fan outlet. This may be used to simulate an outlet duct. The outlet duct friction shall not be considered.
3. Variable supply system may be an auxiliary fan or throttling device.
4. The distance from the exit face of the largest nozzle to the downstream settling means shall be a minimum of 2.5 throat diameters of the largest nozzle.

**Appendix A – Fan test datasheet including all data to be collected.
 Entries denoted by “*” must be calculated using the primary data.**

Test Date		Fan is from	
Fan Manufacturer		Motor Manufacturer	
Fan Model		Motor Model	
Fan Size		Motor Amps/Volts/RPM	
Attachments			
Test Conditions			
Temperature (Wet Bulb), °F		Barometric Pressure, in Hg	
Temperature (Dry Bulb), °F		Corrected Pressure, in Hg	

Time Started	Time Read	# Nozzle	Nozzle Dia. (inch)	Pressure Drop (In. H ₂ O)	Static Pressure (In. H ₂ O)	Airflow (ft ³ /min)	Fan Speed (rpm)	Voltage (V)	Current (A)	Power (W)	VER (ft ³ /min /W)
					0.00	*					*
					0.05	*					*
					0.10	*					*
					0.15	*					*
					0.20	*					*
					0.25	*					*

**MEASUREMENT OF VENTILATION FAN FLOWRATE
USING THE FAN ASSESSMENT NUMERATION SYSTEM (FANS)
Standard Operating Procedure (SOP) A2**

**MEASUREMENT OF VENTILATION FAN FLOWRATE
USING THE FAN ASSESSMENT NUMERATION SYSTEM (FANS)
Standard Operating Procedure (SOP) A2**

Prepared by

Sam M. Hanni and Bill W. Bogan

Reviewed by

Teng-Teeh Lim, Albert J. Heber, and Kenneth D. Casey (Texas A&M Univ.)

Effective Date: December 28, 2007

**PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)
Agricultural and Biological Engineering, Purdue University
West Lafayette, IN 47907**

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions.....	3
4. Health and Safety	3
5. Cautions	3
6. Interferences.....	4
7. Personnel Qualifications.....	4
8. Equipment and Supplies	4
9. Procedures	5
10. Data and Records Management	7
11. Quality Control and Quality Assurance	7
12. References	7
Appendix A. Field Data Sheet for FANS Measurement.....	8

1. Scope and Applicability

- 1.1. A method is needed to spot-measure actual fan capacities in the field, as the performance of a fan deteriorates over time due to dust buildup, belt wear and shutter degradation. This renders published performance curves for new fans unsuitable for accurately determining airflow of fans operating in barns.
- 1.2. This SOP describes the in-situ measurement of ventilation fan flow rate using the Fan Assessment Numeration System (FANS).
 - 1.2.1. The maximum diameter of fan that can be measured with the FANS depends on the size of the analyzer. The FANS that are currently available can test fans up to 30", 48" and 54".
 - 1.2.2. About 30 in. of free space in front of the fan is needed to provide access for the FANS analyzer.

2. Summary of Method

A device for *in situ* fan airflow capacity measurement, called the FANS, previously developed at the USDA-ARS Southern Poultry Research Laboratory, was refined and constructed by University of Kentucky (Gates et al., 2004). It is designed to measure the total airflow rate of a ventilation fan by measuring the air speed across the entire fan cross-section. The FANS incorporates an array of five impeller anemometers to perform a real-time traverse of the airflow entering or exiting an exhaust fan. The five anemometers supply an average airflow velocity across a known cross-sectional area, allowing calculation of the total airflow rate.

3. Definitions

- | | |
|------------|--|
| 3.1. BESS | Bioenvironmental and Structural Systems |
| 3.2. dP | Differential pressure |
| 3.3. FANS | Fans Assessment Numeration System |
| 3.4. PAAQL | Purdue Agricultural Air Quality Laboratory |

4. Health and Safety

- 4.1. Be careful when working with electrical power connections. Instrument repair should only be carried out by trained personnel.
- 4.2. Be careful when working near operational ventilation fans. The high-speed fan blades can be a hazard, especially if they are not protected by fan shutters.
- 4.3. Wear safety glasses when working near operational fans. Avoid wearing loose-fitting or baggy clothing, or dangling jewelry near an operational fan, and tie up long hair.

5. Cautions

- 5.1. Prevent damage by rodents and livestock, especially if the analyzer is stored at the measurement site, by storing it off of the ground, in areas which livestock do not enter.

- 5.2. Store the analyzer away from moisture, manure and mechanical vibration.
- 5.3. Protect instrument from adverse weather, especially precipitation.
- 5.4. Ensure that adequate power is available at the measurement site. Lack of a stable power source can result in the loss of data because of power interruptions. The system requires measurement to start from the top or bottom positions for a complete measurement cycle. If the anemometers are stopped before completing a measurement cycle, use the manual button or program to bring the anemometers to either the top or bottom position before starting another measurement cycle.

6. Interferences

- 6.1. When the field measurement of FANS data covers only a narrow range of static pressures, determining the full fan performance curve is not possible. This can result in greater uncertainty of the calculated airflows, because the degraded fan curve will be estimated based on shape of the undegraded fan curve rather than measured.
 - 6.1.1. To measure the airflow of the exhaust fan over the operating range of dPs, take three measurements, at approximately the minimum, middle and maximum of the static pressure range of the barn. Do this by increasing or decreasing the number of fans that are running, and/or by adjusting the opening(s) of the barn air inlets (baffles, curtains, etc.).
 - 6.1.2. Unless the barn is empty or proper ventilation airflow can be ensured for the comfort of the animals, avoid intervening with the barn's environmental control system to adjust barn static pressure to desired values.
- 6.2. In order to avoid inaccurate airflow data due to blockage, keep all objects, livestock and personnel from moving in front of the fan being tested, to a distance at least two (2) times the fan diameter.
- 6.3. Use of the FANS unit upstream or downstream of a ventilation fan could add pressure drop to the working fan, and hence may reduce performance. However, laboratory test of the FANS at the BESS facility (Gates et al., 2004) suggest that the pressure drop was insignificant.

7. Personnel Qualifications

- 7.1. Personnel should be trained in the use of the instrument and associated software before initiating the procedure.
- 7.2. Personnel should possess a basic understanding of barn ventilation systems, and be able to identify the normal ranges of barn pressure and fan performance.
- 7.3. Personnel should be able to lift 50 lbs.
- 7.4. Each analyst must read and understand Reference 12.1 (Gates et al., 2004) and this SOP before operating the FANS analyzer.

8. Equipment and Supplies

- 8.1. FANS, including appropriate hardware and software (Biosystems and Agricultural Engineering Department, University of Kentucky, Lexington, KY)

- 8.2. Laptop computer for operating the FANS unit and logging the measurement data. The computer must have enough memory (>20 Mb) for data storage, and a CD drive.
- 8.3. RS232 cable – standard 9-pin
- 8.4. Model 27106RS Propeller Anemometer (R.M. Young Company, Traverse City, MI).

9. Procedures

9.1. Installing software

- 9.1.1. Before using the FANS Analyzer for the first time, set up the laptop computer with the necessary software to link with the FANS Analyzer, to control the analyzer and record data. Close down all background applications and programs of the computer.
- 9.1.2. Insert Purdue Calibration Data 2007 Control Interface CD, and install the program by clicking the auto-start program or using the command “setup.exe”.
 - 9.1.2.1. Installation may require an internet connection if the Microsoft.Net framework hasn't already been installed as part of regular updates.
- 9.1.3. The software will automatically install, and an icon (“FANS Interface Application”) will appear on the computer's desktop.

9.2. FANS cart

- 9.2.1. The FANS cart is a metal frame that provides an easy way to move the analyzer from one fan to another.
- 9.2.2. The cart has four trailer jacks (one at each corner) that can be adjusted up and down, and that help in adjusting the height of and leveling of the analyzer (Fig. 1a).
- 9.2.3. Use of the FANS cart is optional; other means (Fig. 1b) can be used to support the analyzer, provided it is kept level.



(a)



(b)

Figure 1. Measuring fan airflow rate using a FANS analyzer positioned on the outlet side of a fan with the aid of the FANS cart (a) and on the fan inlet, supported by cinderblocks (b).

9.3. Operation

- 9.3.1. Supply power to the FANS and laptop computer with stable 110 VAC power source.
 - 9.3.1.1. Switch on the unit by turning the power switch to the on position.
 - 9.3.1.2. Operate the unit manually by toggling the up and down switch to make sure the motor is moving the anemometers upward or downward freely.
- 9.3.2. Install impellers onto the anemometers.
 - 9.3.2.1. The impellers cannot be installed at the bottom position due to limited space; the holder must be at least six inches from the bottom.
 - 9.3.2.2. Make sure that the numbers on the impellers match those on the anemometers.
 - 9.3.2.3. Reposition the anemometer holder back to either the top or bottom position.
- 9.3.3. Center the FANS, both horizontally and vertically, in front of the desired fan, either at the exhaust (Fig. 1a) or inlet (Fig. 1b) side. If possible, enclose the entire cross-sectional area of the fan to make sure all airflow goes through the FANS for accurate measurement. Use the cart's wheels to adjust position horizontally, and the jacks to adjust vertically.
- 9.3.4. Connecting the computer and FANS.
 - 9.3.4.1 Double click on the "FANS Interface Application" icon.
 - 9.3.4.2 Find the serial number on the FANS Analyzer (ex. FANS 48-0015)
 - 9.3.4.3 In the FANS Interface Application software, click "Edit" then "Serial Number".
 - 9.3.5.3.1 This will give a pull-down list of serial numbers. Select the number of the analyzer that is being used.
 - 9.3.5.3.2 Once the serial number is selected, the FANS Interface Settings and BESS Calibration Information will be automatically set.
 - 9.3.4.4 Data acquisition and automated control are provided through the RS232 interface.
 - 9.3.4.4.1 Use a standard 9-pin serial cable to connect the unit to the computer.
 - 9.3.4.5 In the FANS Interface Application software, click "Edit" then "Communication Port".
 - 9.3.4.5.1 This will give a pull-down list of communication ports. Select the proper port.
 - 9.3.4.6 In the FANS Interface Application software, click "File" then "Connect to FANS".
 - 9.3.4.6.1 Data will not be read unless the FANS Analyzer has power and is turned on.
 - 9.3.4.6.2 If the FANS is connected, the "Status" should read "Connected".
- 9.3.5. Bring the anemometer bar back to either the top or bottom position.
- 9.3.6. Start the measurement by clicking "Run Test".
 - 9.3.6.1. It will take 186 s to complete one airflow measurement. Data will scroll as it is being collected, and will automatically save to a file when the measurement is complete.
 - 9.3.6.2. Record the sampling location, barn and fan numbers, date and time, and other details such as fan operating dP, weather, and other observations in the field sampling data sheet (Appendix A).
 - 9.3.6.3. Record the measurement data (speed and flow rate) output by the program.
 - 9.3.6.4. Repeat the measurement. If the difference between the two measurements is >5%, despite a constant dP, repeat measurement until the difference (standard deviation) of all measurements is $\leq 5\%$.

- 9.3.7. It is important to note all observations and changes that could affect the fan dP, including changes in number of fans operating, changes in wind direction and velocity, and any other factors that could affect the fan airflow.
- 9.4. When measurements are completed at a particular fan and the computer and power must be disconnected from the FANS, make sure to click “File” then “Disconnect Fans”. Move the equipment to the next fan, and repeat Steps 9.3.3 through 9.3.7.
 - 9.4.1. Apply silicone grease to the chain, bearings, all-thread support, and other mechanical parts of the FANS to make sure they are well lubricated. The necessary lubrication frequency will depend on several factors, including dust levels, relative humidity, and how the FANS is used. The grease should be added at the operator’s discretion.
 - 9.4.2. When sampling is completed for the day, disconnect the power, and place all items in a protected location, such as the OFIS (On-Farm Instrument Shelter).

10. Data and Records Management

- 10.1. Maintain all field sampling records in a worksheet of the electronic field notes spreadsheet that is designated for this method. Supplement this electronic record with a bound record book designated for the method, which should also contain bound copies of the electronic record.
- 10.2. Manage all data according to SOP B5.
- 10.3. Document all of the data and information on field data sheets, and site logbooks with permanent ink, or in electronic field notes. Initial and date all corrections.

11. Quality Control and Quality Assurance

- 11.1. At the beginning and end of each set of field measurements, perform a “zero airflow” measurement to make sure the program reads an insignificant zero value. Do this by operating the FANS with no impellers attached. The zero reading should be less than 3 m³/min (106 ft³/min).
- 11.2. Back up the FANS folder at the end of each day on which field measurements are performed, to ensure an extra copy of the measurement data. The measurement data logged by the Anemometer2 program contains the date and time, mean velocity and flow rate for each of the measurement run, and mean values for each anemometer.
- 11.3. Calibrate each FANS unit annually at the UIUC (University of Illinois at Urbana-Champaign) BESS Lab.

12. References

- 12.1. Gates, R.S., K.D. Casey, H. Xin, E.E. Wheeler, and J.D. Simmons. 2004. Fan Assessment Numeration System (FANS) design and calibration specifications. Transactions of ASAE 47(5): 1709-1715.
- 12.2. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.

Appendix A. Field Data Sheet for FANS Measurement.

Project _____
FANS Measurement Field Sheet

Date	_____
Operator	_____
Weather	_____
Fan location	_____
Site/Building	_____

Run	Time start	Time stop	Flow rate	Speed	Notes

Fan location	_____
--------------	-------

Run	Time start	Time stop	Flow rate	Speed	Notes

Fan location	_____
--------------	-------

Run	Time start	Time stop	Flow rate	Speed	Notes

VENTILATION FAN AIR VELOCITY MEASUREMENTS USING

OPEN IMPELLER ANEMOMETRY

Standard Operating Procedure (SOP) A3

**VENTILATION FAN AIR VELOCITY MEASUREMENTS USING
OPEN IMPELLER ANEMOMETRY
Standard Operating Procedure (SOP) A3**

Prepared by

Sam M. Hanni and Bill W. Bogan

Reviewed by

Teng-Teoh Lim and Albert J. Heber

Effective Date: November 6, 2007

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions	3
4. Health and Safety	4
5. Cautions	4
6. Interferences	4
7. Personnel Qualifications.....	5
8. Equipment and Supplies	5
9. Procedures	5
10. Data and Records Management	Error! Bookmark not defined.
11. Quality Control and Quality Assurance	9
12. References.....	11

1. Scope and Applicability

- 1.1. This SOP describes monitoring of wall and pit ventilation fan air speeds and operational status using an impeller anemometer.
- 1.2. This method applies to the measurement of air velocities in locations where cyclonic flow conditions exist and temperatures range from 10°C and 40°C (barn exhaust air)
- 1.3. This SOP is specific to the R.M. Young Company's Model 27106RS Impeller Anemometer.
 - 1.3.1. This anemometer will be used for monitoring ventilation fans ranging from 18 to 54 in. in diameter.
 - 1.3.2. The diameter of this anemometer is 7 in.; thus, it will measure airflow velocity of a small fraction of the fan's cross-sectional area, ranging from 16% on an 18-in. fan to 2% on a 54-in. fan.
 - 1.3.3. The anemometer can be placed upstream or downstream of the fan.
 - 1.3.4. With the specifications in this SOP, this anemometer has a lower threshold of 1 m/s, and an upper threshold of approximately 45 m/s.
 - 1.3.5. Measurements are taken continuously.

2. Summary of Method

The Model 27106RS anemometer is equipped with a four-blade helicoid impeller, which drives a miniature tach-generator transducer. The generator converts the rotation of the impeller into a DC voltage. Output voltage from this generator is directly proportional to the axial component of the air speed. Signal polarity indicates direction of impeller rotation. The instrument should be oriented with the impeller facing the predominant flow of air being measured. The tachometer generator output is set at the factory for 500 ± 2 mV at 1800 rpm. The pitch of the impeller is 0.00478 m/s per rpm, with an accuracy of $\pm 1\%$. Data collected from anemometers is logged by the AirDAC software program (SOP B2). While the method in this SOP measures air velocity directly, the fan airflow rate is calculated based on a velocity vs. airflow curve for the anemometer and the fan. To develop this curve, the anemometer is installed within a fan, and tested with a low-turbulence wind tunnel (SOP A1). The curve is then verified in field with a calibrated portable fan tester (Fans Assessment Numeration System, FANS) that consists of multiple traversing impeller anemometers (SOP A2). Sections 9.1 through 9.4 of this SOP describe procedures for using the anemometer to measure airflow velocity on-site, while Section 9.5 covers use of the anemometers in generating the velocity vs. airflow curve. Procedures for initial ("benchmarking") performance testing of anemometers, and for periodic performance audits are also included.

3. Definitions

- | | |
|-------------|--|
| 3.1. AirDAC | Data Acquisition and Control Software |
| 3.2. PAAQL | Purdue Agricultural Air Quality Laboratory |
| 3.3. QAPP | Quality Assurance Project Plan |
| 3.4. rpm | Revolutions per minute |
| 3.5. SOP | Standard operating procedure |

- 3.6. Transfer function The ratio of air speed to anemometer rotation rate (in rpm)

4. Health and Safety

- 4.1. Make sure that the ventilation fan in which the anemometer is being placed is turned off manually before anemometer installation, and during all maintenance.
- 4.2. Be careful when working with electrical power connections. Repair of instruments should only be carried out by trained personnel, and in accordance with procedures detailed in the instrument manual.

5. Cautions

- 5.1. Connecting the wires to cable connectors on the impeller anemometer improperly could cause a positive signal when the impeller rotates counterclockwise, which is not desired (Section 9.6.2.2.1).
- 5.2. Avoid placing the anemometer too close to the center (hub) of the ventilation fan. The region is often characterized by very low air velocity, and sometimes even reverse flow. Placing the anemometer close to this region will reduce the rotational speed of the anemometer. Previous experience has shown that the ideal location on most fans is approximately $\frac{1}{3}$ the distance from the fan housing wall to the outer edge of the fan hub.
- 5.3. When using anemometers to monitor multiple fans of the same size and model, position the anemometer at the same spot on each fan, and the same distance from the fan. Record this position and distance for each anemometer.
- 5.4. Worn or improperly functioning fan shutters can change the velocity profile of the fan, which will affect the readings of the anemometer.
- 5.5. Cleaning and maintaining the shutters can affect fan capacity and velocity profile.
- 5.6. Damage to the fan housing can change the velocity profile.
- 5.7. Before conducting any maintenance on a fan that is being monitored by an anemometer, remove the anemometer to avoid damage.
- 5.8. Avoid installing an anemometer too far away from the fan on the inside of the barn, as this could affect worker activity in the barn, or result in the anemometer being damaged or knocked out of alignment.
- 5.9. The most common problem with the Model 27106RS is partial or complete bearing failure. The bearing failure usually leads to increased friction, which results in reduced signal output and an underestimation of airflow speed. This problem is diagnosed through the torque test described in Section 11.1.1.
- 5.10. When the tach-generator output becomes erratic (usually due to brush failure) or begins to show signs of bearing failure (high torque), the entire generator assembly should be removed and replaced

6. Interferences

- 6.1. Dust collected on the anemometer could reduce its rotational speed. Check for dust accumulation, and clean anemometers as necessary.

- 6.2. Make sure the signal wires from the anemometer are firmly attached to the anemometer support and tidy to avoid being blown loose over time.
- 6.3. Wind speed and wind currents around the barn can affect rotation of impeller. Therefore locate the impeller anemometer close to the ventilation fan and make sure that the outside wind cannot affect the direction or speed that the impeller is turning.
- 6.4. Moisture can cause the bearings to rust, and can lead to a bad connection between the impeller anemometer and the DAQ (Data acquisition) hardware.

7. Personnel Qualifications

- 7.1. Personnel should be trained in the use of the instrument, including the associated calibration equipment, before initiating the procedure.
- 7.2. Each analyst must read and understand the entire instrument manual and this SOP before operating the instrument.

8. Equipment and Supplies

- 8.1. Impeller anemometer (Model 27106RS, R.M. Young Company, Traverse City, MI)
- 8.2. Anemometer torque disc (Model 18310, R.M. Young Company, Traverse City, MI)
- 8.3. Anemometer rotational calibration unit (Model 18802, R.M. Young Company, Traverse City, MI)
- 8.4. Voltage & resistance meter
- 8.5. Stop watch
- 8.6. 5-ft. steel fencing post and post driver
- 8.7. Hose clamps
- 8.8. Level
- 8.9. Screwdriver
- 8.10. Pocket knife
- 8.11. Flange bearings (part # 27122)
- 8.12. Tach generator
- 8.13. Soldering kit

9. Procedures

- 9.1. Siting
 - 9.1.1. Always place the anemometer shaft parallel to the ventilation fan shaft. For example, if the fan is mounted into the wall with a downward angle, the anemometer should be mounted facing upwards if it is mounted outside the fan, or downwards if it is mounted inside the fan.
 - 9.1.2. When placing the anemometer on the inlet side of ventilation fan, face the impeller toward the fan blades. The impeller should be at least 4 in. away from the fan blades.
 - 9.1.3. When placing the anemometer on the exhaust side of the fan, face the impeller towards the fan blades, with the anemometer impeller at least 4 in. away from the ventilation fan blades.

- 9.1.4. Record the distance and direction of the anemometer from the fan blades and the shaft of the fan. If necessary, include a drawing detailing the location in the site monitoring plan. See Section 5.3 if monitoring more than one fan of the same size and model.
- 9.1.5. If possible, protect the anemometer from wind by placing it within the fan housing.
- 9.1.6. Depending on the location of the fan guards, the anemometer can be placed upstream or downstream of the fan guard.
- 9.2. Installing anemometer at outlet side of ventilation fan
 - 9.2.1. Turn off the ventilation fan before any work is started.
 - 9.2.2. Using a post driver, drive a steel post into the ground until the stabilizing blades of the post are completely buried (Fig. 1).



Figure 1. Model 27106RS Impeller Anemometer installed on a post on the exhaust side of a ventilation fan (Hoff et. al. 2004)

- 9.2.3. Place the anemometer at the proper monitoring location. If the impeller is to be positioned between the fan guard and fan blades, remove the impeller from the anemometer shaft and housing. Do not place the anemometer in line with the hub of the fan (Section 5.2).
- 9.2.4. Secure the shaft housing and generator housing to the steel post using hose clamps.
- 9.2.5. If the impeller was removed, replace it onto the shaft.

- 9.2.6. Restart the ventilation fan.
- 9.3. Installing anemometer at inlet side of ventilation fan
 - 9.3.1. Shut off the ventilation fan before any work is started.
 - 9.3.2. Many ventilation fans have a fan guard or shutters on the inlet side.
 - 9.3.3. If there are shutters, remove and replace with a fan guard if the ventilation fan is kept running throughout the year.
 - 9.3.4. If the fan is not a winter fan and the shutter cannot be replaced, cut a small section (1 in.) out of one of the shutter blades, and install the anemometer. The objective is to minimize the leakage of the shutter when the anemometer is installed through the shutters.
 - 9.3.5. Connect the anemometer shaft to the fan guard or shutter if one of these is located such that it would place the anemometer in the desired location, If not, install an additional supporting pole. Use hose clamps or similar rigid means to attach the anemometer to the support.
 - 9.3.6. Make sure that the anemometer shaft is parallel with the fan shaft (Section 9.1.1). Do not place the anemometer directly in line with the fan hub (Section 5.2).
 - 9.3.7. Restart the ventilation fan.
- 9.4. Operation and data collection
 - 9.4.1. The anemometer analog signals (voltages) are recorded by the data acquisition system, as specified in SOP B2. Data will be one-minute averaged for recording purposes.
 - 9.4.2. The DAS (Data acquisition software) will also convert the output signal into air velocity.
- 9.5. Estimating fan airflow rate based on anemometer air velocity measurement
 - 9.5.1. If possible, remove used fan(s) from the measurement site and test their performance with a low-turbulence wind tunnel (SOP A1). If this is not possible, use a new fan of the same manufacturer and model.
 - 9.5.2. When testing fan performance, install the anemometer within the fan housing and record velocity readings. It is very important that the anemometer position is carefully chosen as discussed above, and is the same as that which will be used in the field.
 - 9.5.3. Correlate the measured anemometer velocities and wind tunnel flow rates to develop fan performance curves based on anemometer velocity data (Fig. 2).
 - 9.5.4. Measure *in-situ* fan airflow capacities with a calibrated portable FANS analyzer (SOP A2). Use these measured values, together with the anemometer velocities obtained at the same time, to update the relationship in Fig. 2. This field spot-check is necessary because differences exist for each ventilation fan, and their capacities can change over time.

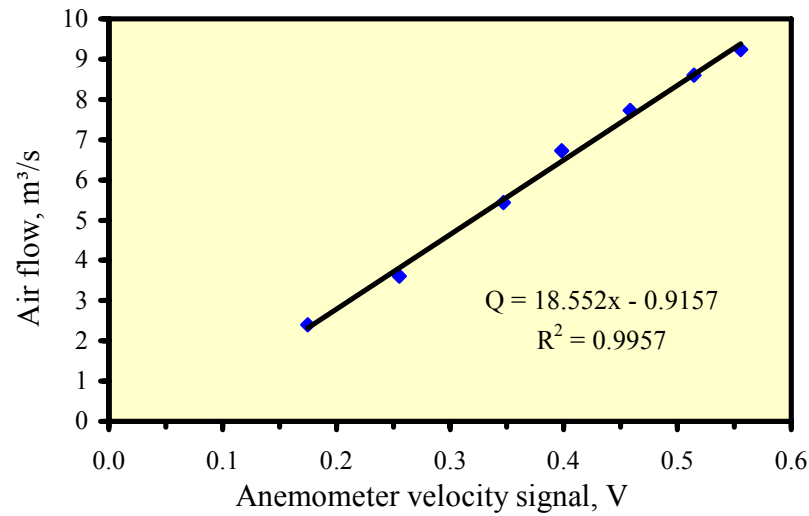


Figure 2. Anemometer velocity vs. fan airflow rate, based on wind tunnel measurements.

9.5.5. Based on the comparison of field spot-checks and wind tunnel data, develop an individual fan performance curve (anemometer velocity vs. airflow rate) for each fan that is monitored by a vane anemometer, to determine airflow rate based on the single-spot velocity measurement.

9.6. Maintenance

9.6.1. Replacement of bearings

9.6.1.1. Removing old bearings

9.6.1.1.1. Remove impeller from anemometer.

9.6.1.1.2. Unthread and separate shaft housing assembly from generator housing.

9.6.1.1.3. Loosen set screw on shaft collar/coupling disk, and remove from impeller shaft.

9.6.1.1.4. Slide impeller shaft through both bearings and out of housing.

9.6.1.1.5. Remove front bearing dust shield from housing.

9.6.1.1.6. Gently pry front and rear bearings out of housing using the edge of a pocket knife or a sharp screw driver.

9.6.1.2. Installing new bearings

9.6.1.2.1. Gently insert front bearing into housing.

9.6.1.2.2. Install front bearing dust shield through front bearing and into housing.

9.6.1.2.3. Carefully slide impeller shaft through front bearing and into housing.

9.6.1.2.4. Slide rear bearing over impeller shaft and gently push it into housing.

9.6.1.2.5. Place shaft collar/coupling disk on impeller shaft.

9.6.1.2.6. Thread shaft housing assembly into generator housing and tighten firmly.

9.6.1.2.7. Check bearing torque to confirm it is within specifications.

9.6.2. Tach-generator replacement

9.6.2.1. Removing old generator assembly

9.6.2.1.1. Remove impeller from anemometer.

- 9.6.2.1.2. Unthread generator housing collar. Remove generator housing from sensor connector and generator assembly.
- 9.6.2.1.3. Note position of generator wires on sensor connector pins. Unsolder wires from pins and remove old generator assembly.
- 9.6.2.2. Installing new generator assembly
 - 9.6.2.2.1. Solder wires of new generator assembly onto proper connector pins. Verify correct polarity: Counterclockwise (CCW) rotation should produce a negative output voltage.
 - 9.6.2.2.2. Slide generator housing over generator assembly. Tighten housing collar onto connector threads.
 - 9.6.2.2.3. Check bearing torque to confirm it is within specifications.

10. Data and Records Management

- 10.1. Maintain laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
- 10.2. Manage all data according to SOP B5.
- 10.3. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes.

11. Quality Control and Quality Assurance

- 11.1. Initial inspection
 - 11.1.1. Torque test
 - 11.1.1.1. Keeping anemometer in the horizontal orientation, remove impeller, and replace with Model 18310 torque disc (Fig. 3).



Figure 3. Model 18310 Impeller Torque Disc (R.M. Young 2000).

- 11.1.1.2. Use torque disc to measure anemometer torque in both directions (clockwise and counterclockwise). Using a combination of black nylon screws (0.1 g) and pan-head stainless steel screws (1.0 g), establish weights at the various radii on the torque disc (see figure on Pg.7 of RM Young Manual 18860-90) to create the desired torque. Hold torque disc with the weights in a horizontal line (as in Fig. 3), then release the disc. Disc should rotate freely downwards.
- 11.1.1.2.1. This provides a check of the anemometer's bearing conditions. Failure to freely rotate downwards indicates degraded bearings
- 11.1.1.2.2. The torque disc allows verification of the anemometer bearing torque with 0.1 g-cm resolution.
- 11.1.1.2.3. The torque disc can also be used for simple and accurate pass/fail checks.
- 11.1.1.2.4. For a new anemometer, the acceptable threshold is 1.5 g-cm. This corresponds to a stainless steel screw at the 1-cm radius, plus a nylon screw at the 5-cm radius. For a used anemometer, in order to maintain a threshold of 1.0 m/s, the acceptable maximum torque value (from Pg. 9 of the manual) is 2.5 g-cm, which corresponds to a stainless steel screw at the 2-cm radius, plus a nylon screw at the 5-cm radius.
- 11.1.2. Measurement of transducer output
- 11.1.2.1. Attach the Model 18802 Anemometer Drive unit (Fig. 4) to the anemometer, and connect a voltmeter to the "A" and "B" output pins of anemometer. The Model 18802 is a selectable-speed unit which can be run at any speed between 100 and 10,000 rpm.
- 11.1.2.2. Run the Model 18802 at the following speeds, and record its voltage output:
- 300 rpm
 - 1800 rpm
 - 3600 rpm
- 11.1.2.3. Repeat for the opposite rotational direction.



Figure 4. Model 18802 Anemometer Drive (R.M. Young 2000).

11.1.2.4. Factory-specified voltage outputs for the Model 27106RS are as follows:

Rate of Rotation (rpm)	Signal Output (mV)
300	83.3
1800	500
3600	1000

11.1.2.4.1. Deviation of more than ± 6 mV at any rate of rotation indicates possible problems with the unit's signal conditioner, transmitting system, averaging system and/or recording system. A unit in this condition should be returned to the manufacturer for repair.

11.2. Periodic inspection and maintenance

11.2.1. Perform torque tests according to the frequency specified in the QAPP for the study.

11.2.1.1. Replace the bearings if the measured torque is greater than the maximum allowed, and repeat the torque test.

11.2.2. Measure transducer output according to the frequency specified in the QAPP.

11.2.2.1. Deviation(s) of more than ± 6 mV indicate possible problems with the anemometer signal conditioner, transmitting system, averaging system and/or recording system. Any unit in this condition should be returned to the manufacturer for repair or replaced with a new unit.

12. References

- 12.1. Anemometer Manual. 2002. Operations Manual for Model 27106RS Impeller Anemometer (PN 27106R-90). R.M. Young Co., Traverse City, MI.
- 12.2. Wind System Calibration. 2003. Manual PN18860-90. RM Young Co, Traverse City, MI.
- 12.3. Calibration Accessories 2000. R.M. Young C. Traverse City, MI. Online at www.youngusa.com/18860.pdf. Accessed 7/6/2006.
- 12.4. SOP A1. 2006. Laboratory Testing of Ventilation Fan Performance. Standard Operating Procedure A1. Purdue Ag Air Quality Lab.
- 12.5. SOP A2. 2006. Measurement of Ventilation Fan Flowrate Using the Fan Assessment Numeration System (FANS). Standard Operating Procedure A2. Purdue Ag Air Quality Lab.
- 12.6. SOP B2. 2006. Data Acquisition and Control Software (AirDAC). Standard Operating Procedure B2. Purdue Ag Air Quality Lab.
- 12.7. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 12.8. Hoff, S.J., D.S. Bundy, M.S. Huebner, B.C. Zelle, L.D. Jacobson, B.P. Hetchler, V.J. Johnson, D.R. Schmidt, P.R. Goodrich, A.J. Heber, J.Q. Ni, T.T. Lim, P.C. Tao, Y. Zhang, J. McClure, S. Jerez, M. Roberts, J.A. Koziel, B.H. Baek, A. Balota, J.P. Spinhirne, J.M. Sweeten, D.B. Beasley, G.R. Baughman, and R. Munilla. 2004. Real-time ventilation measurements from mechanically ventilated livestock buildings for emission rate estimations. ASAE Paper No. 044178. American Society of Agricultural and Biological Engineers, St. Joseph, MI.

BARN VENTILATION RATE ESTIMATION
Standard Operating Procedure (SOP) A4

BARN VENTILATION RATE ESTIMATION

Standard Operating Procedure (SOP) A4

Prepared by

**Teng-Teoh Lim, Matthew J. Darr (Ohio State University), and
Lingying Zhao (Ohio State University)**

Reviewed by

Bill W. Bogan, Albert J. Heber and Erin Cortus

Effective Date: November 6, 2006

Rev. 1.0 Effective Date: March 17, 2008

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability.....	3
2. Summary of Method.....	3
3. Definitions.....	4
4. Health and Safety.....	5
5. Cautions.....	5
6. Interferences.....	5
7. Personnel Qualifications.....	6
8. Equipment and Supplies.....	6
9. Procedures.....	6
10. Data and Records Management.....	10
11. Quality Control and Quality Assurance.....	10
12. References.....	10

1. Scope and Applicability

- 1.1. This method determines the total ventilation rate of a mechanically-ventilated livestock barn. The one-minute barn ventilation rate is the summation of the individual fan airflow rates over that minute.
- 1.2. A mechanically-ventilated barn is often ventilated in multiple ventilation stages, with each stage consisting of one or more fans and fan locations.
- 1.3. This method applies to both single- and variable-speed ventilation fans.
- 1.4. The method accounts for fan performance degradations, which generally range from 0 to 50% decrease in airflow rate. Degradation is generally higher for fans in poultry barns than in dairy and swine barns, due to the much higher dust levels in the former.

2. Summary of Method

The ventilation rate of a mechanically-ventilated livestock building is the sum of the airflow of all individual fans operating at any given time. The airflow rate of a single fan is a function of its operational status, its designed performance, its degradation from that optimal performance, and the differential pressure of the building. Building static pressure will be continuously recorded (SOP A5). The operational status (operating time, %) of individual fans will be monitored using vibration sensors (SOP A7) (Ni et al., 2005), fan speed (rpm) sensors (SOP A11), current switches, and/or impeller anemometers (SOP A3). The operational status of each fan stage (which may consist of one or more individual fans) via auxiliary contacts of fan motor control relays (SOP S4) will provide a cross-reference set of data in most cases. Ventilation stage monitoring is necessary for barns where each fan is not monitored for operation status. Open impeller anemometers will, in general, be placed on selected primary representative exhaust fans, and on one representative fan for every other fan model employed at the site. These anemometers provide further information regarding fan performance. The fan performance curves will be obtained or determined for each fan model. If possible, this will involve testing an existing fan of each model (one removed from the barn) by a fan test facility (SOP A1). Published fan curves are also available for most fan models.

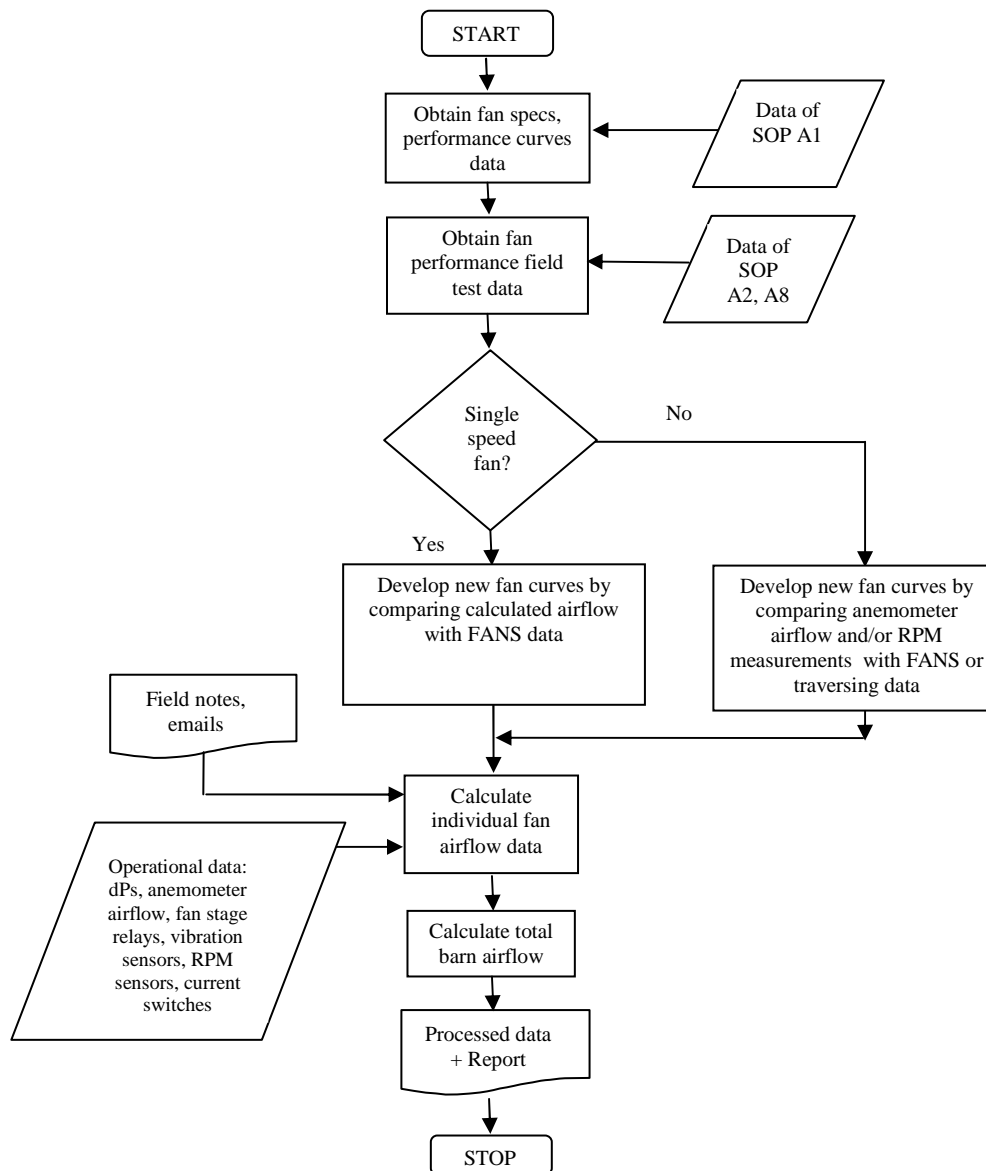
Individual fan airflow capacities will then be measured and verified in the field, so that the baseline fan performance curves for each ventilation fan can be properly adjusted to reflect field conditions and fan degradation. If possible, this will be done with a calibrated portable fan tester (FANS, Fans Assessment Numeration System) that consists of multiple traversing impeller anemometers (SOP A2; Gates et al., 2004), as a transfer standard. If the FANS cannot be used, fan airflow will be determined with a manual traversing method (SOP A8).

For single-speed fans, the fan airflow rate is calculated based on the status of the fans (on/off and operation time in % of a minute), calibrated individual fan performance curves, and the building differential pressure.

For variable-speed fans, the rpm measurement (SOP A11) or the open-impeller velocity measurement (SOP A3) will be used in conjunction with the status of the fans (on/off and

operation time in % of a minute), calibrated individual fan performance curves, and the building differential pressure, to calculate the fan airflow rate.

The ventilation rate for the barn as a whole is then determined by summing the individual airflow rates of all fans in the barn. The flow chart below summarizes the method for Barn Ventilation Rate Estimation.



3. Definitions

- | | |
|-----------|--|
| 3.1. atm | 1 atmosphere (101,325 Pa) |
| 3.2. BESS | Bioenvironmental Systems and Simulations |
| 3.3. dP | Differential pressure |

- 3.4. FANS Fans Assessment Numeration System
- 3.5. PAAQL Purdue Agricultural Air Quality Laboratory
- 3.6. QAPP Quality Assurance Project Plan
- 3.7. SOP Standard operating procedure
- 3.8. STP Standard temperature and pressure

4. Health and Safety

- 4.1. Be careful when working near ventilation fans. The high-speed fan blades can be a hazard, especially if they are not protected by fan shutters. Safety glasses should always be worn while in the presence of an operating fan. Do not wear loose-fitting or baggy clothing, or dangling jewelry around an operational fan, and put up or secure long hair.

5. Cautions

- 5.1. Obtain the correct fan test data from the fan manufacturer. Since there are many similar fans, it is very important to provide the correct fan information, including the size, model number, motor details, shutter type, fan guard, housing, etc.
- 5.2. Ideally, *in situ* FANS measurements should be conducted at several differential pressures (dPs), which should cover the entire operating range. However, unless it is possible to intervene with the environmental control system to adjust barn dP to desired values, it may only be possible to conduct the FANS measurement replications over a narrow range of dP or a small segment of the fan performance curve. The rest of the curve is estimated.
 - 5.2.1. Producer permission must be granted for intervention of the barn environmental control system.
 - 5.2.2. Exceptions to this may include empty barns, or cases when proper ventilation airflow can be ensured for the comfort of animals during the FANS measurement.
- 5.3. When the field measurement of FANS data covers only a narrow range of the operating pressure, comparisons with the manufacturer's fan performance curve are somewhat limited, and result in greater uncertainties in airflow rate calculations.
- 5.4. Dust collected on the impeller anemometer could reduce its rotational speed, and affect air velocity measurement accuracy. The impellers need to be cleaned and checked periodically according to the frequencies as stated in the Quality Assurance Project Plan (QAPP) for the particular project.

6. Interferences

- 6.1. Fan performance can be affected by dust accumulated on shutters, belt wear, and damage to the shutters and/or fan housing.
- 6.2. Strong wind around the barns can impose pressure onto the fans; however, these effects should be measured by the differential pressure sensors and the impeller anemometers.
- 6.3. Fan maintenance, such as changing or tightening the belt, or cleaning the shutter and/or fan housing, can affect fan performance. Obtain maintenance records from the farm manager to determine whether reevaluation of the fan capacities is necessary (SOP S1).

- 6.4. Barn managers sometimes change ventilation stage strategies. Follow SOP S1 to ensure that any such changes are reported to the study personnel. Cross-check the individual fan operation data (from anemometers or vibration sensors) with the ventilation stage data (from contactor relays) before performing airflow calculations. If necessary, update the number of fans controlled by each ventilation stage, so that the barn airflow will be calculated based on the new strategy.
- 6.5. Use of the FANS unit upstream or downstream of a ventilation fan could add a pressure drop to the working fan, and hence may reduce performance. However, both laboratory tests of the FANS at the University of Illinois Bioenvironmental Systems and Simulations (BESS) facility (Gates et al., 2004) and field measurements of layer barn ventilation fans (Lim et al., 2003) suggest that this pressure drop is insignificant.

7. Personnel Qualifications

- 7.1. Personnel should be knowledgeable about ventilation stage control of animal buildings, fan performance curves, and ventilation needs of animal facilities.
- 7.2. Personnel should be trained in the use FANS, and all instruments, and software mentioned in this SOP.

8. Equipment and Supplies

- 8.1. Commercial spreadsheet software (e.g. Microsoft Excel), and custom software: Air Quality Data Acquisition and Control Software (AirDAC) Raw Data Inspection (SOP B3), and CAPECAB Data Processing Software (SOP B6).
- 8.2. Fan Assessment Numeration System (FANS), including appropriate hardware (notebook PC and data acquisition card, cable, etc) and software (SOP A2)
- 8.3. SETRA Model 260 static pressure sensor (Setra Systems, Boxborough, MA) (SOP A5) or equivalent
- 8.4. Ventilation fan operation monitoring devices such as electromechanical relays (SOP S4), vane anemometers (SOP A3), vibration sensors (SOP A7), rpm sensors (SOP A11) and/or current detectors

9. Procedures

- 9.1. Gather fan information
 - 9.1.1. Obtain specifications of each ventilation fan (Table 1).
 - 9.1.2. Gather existing fan performance data from the fan manufacturer, or from the BESS fan test reports.
 - 9.1.3. Learn and verify the barn's environmental control systems, including the staging details of all ventilation fans. Consult the Site Monitoring Plan to ensure that they have not changed since this was prepared.
- 9.2. Ventilation fan performance evaluation in the laboratory
 - 9.2.1. Conduct lab tests of each fan model in the barn with a fan test facility (SOP A1). Measure the performance in new or refurbished condition with the existing housing, shutters and all other accessories included.

Table 1. Fan specifications.

Parameter	Model 1	Model 2	Model 3
Manufacturer			
Fan Model			
# of this Model in Barn			
Serial Numbers			
Diameter, m			
Shutter (Yes/No, Type)			
Guard (Yes/No, Type)			
Shroud (Yes/No, Type)			
Cone (Yes/No, Type)			
Single Speed?			
Motor Manufacturer			
Motor Model			
Motor Serial Number			
Motor Size, Hp			
No Load Current			
Design Load Current			
Airflow at 0 iwg, cfm			
Airflow at 25 iwg, cfm			

9.2.2. If possible, remove the existing barn fan(s) from the monitored barn(s), and test their performance with a fan test facility to determine degraded fan performance (SOP A1). Conduct tests before and after refurbishment by cleaning and replacing worn parts (e.g. belts, shutters, etc.).

9.2.2.1. Using Microsoft Excel, fit a polynomial equation to the tested (as found or degraded) data to produce a curve, and compare this with the published or baseline data for new fan (Fig. 1). These equations will be fan-specific.

9.2.2.2. Verify that the polynomial equation is theoretically sound over the expected range of dP measurements for the barn. This may include positive dP values.

9.2.2.3. The equations representing the fan curves may be third-order (or higher) polynomial equations, and should have high R-squared values when compared with the measured data. For example, the equations used to calculate airflow rate of the fan in Fig. 1 included the published fan curve and corresponding correction factor of each fan or fan stages (Lim et al., 2003):

$$V = -1.64 \times 10^{-7} P^4 - 4.09 \times 10^{-6} P^3 - 5.09 \times 10^{-5} P^2 + 0.054 P + 11.433 \quad (1)$$

$$K_1 = 1.88 \times 10^{-6} P^3 + 5.11 \times 10^{-5} P^2 + 2.78 \times 10^{-3} P + 0.759 \quad (2)$$

$$Q_1 = S V K_1 \quad (3)$$

Where:

V = volumetric ventilation rate of published fan curve, m³/s

P = building static pressure, Pa.

K₁ = correction factor of ventilation fan1

Q_1 = corrected fan airflow rate for fan 1 m^3/s .

S = Operation status, fraction of time unit, which is usually one minute.

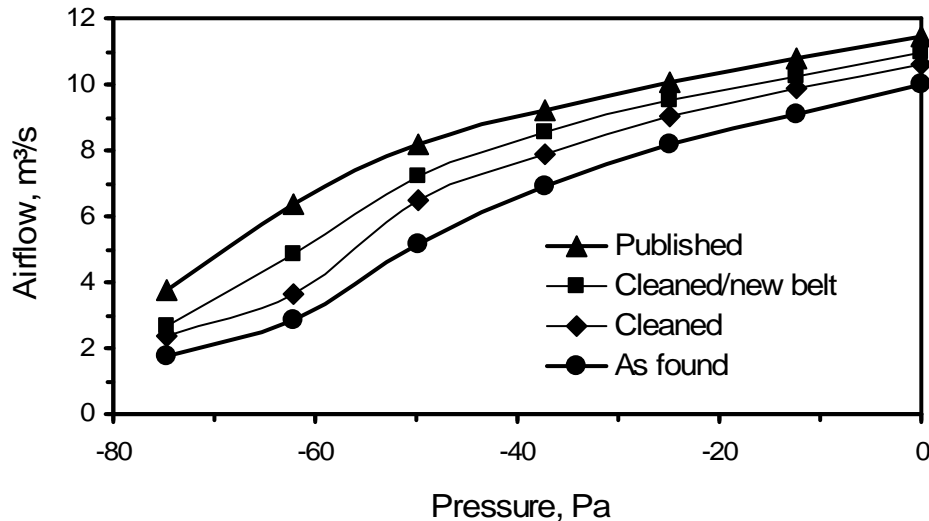


Figure 1. New (Published) and degraded (as found) fan curves. Fan was tested as found, after cleaning, and after replacing fan motor belt (Lim et al. 2003).

9.2.3. Calibrate the FANS with the fan model(s) to be tested in the field (SOP A1).

9.3. Ventilation fan performance evaluation in the field

9.3.1. Conduct fan tests in the field using a calibrated FANS (SOP A2) or the air velocity traverse method (SOP A8) to determine actual *in situ*, degraded fan curves or a degradation correction variable (K_1 , Eq. 2).

9.3.1.1. For single-speed fans, fit a polynomial equation to the fan performance curves for each fan. If the measurement represents only a small segment of the dP range, average the differences between the FANS data in the field and those predicted by the degraded fan curve determined by the lab test facility, and use the value as an offset to the fan curve. Develop a new fan curve with the adjusted constant value in the as-found equation (Fig. 2).

9.3.1.2. For variable-speed fans, compare the FANS data with the airflow determined with the open anemometer or other fan speed method. Determine the needed offset (similar to Section 9.3.1.1) in the equations for estimating fan airflow.

9.4. Variables and information needed for airflow rate calculations for individual fans

9.4.1. Operational status (on/off) of all fans, based on ventilation stage status data, impeller anemometers, vibration sensors, current switches, and/or rpm sensors. If more than one measurement method is used for a fan, the data from one method can be used to cross-check the data from another method.

9.4.2. Verify that measured dP values are correct (SOP A5), and that they correspond to the location of the fan(s).

9.4.3. Study all field notes, log books, and emails to learn about any fan, and operational and fan speed sensor malfunctions.

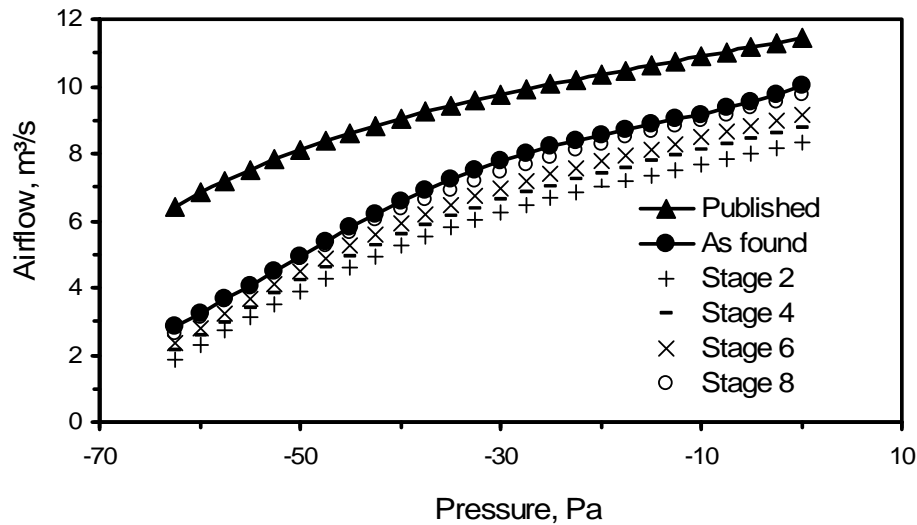


Figure 2. Published fan curve as compared with as-found fan curves for selected ventilation stages (Lim et al. 2003).

9.5. Ventilation rate calculations

- 9.5.1. Calculate the minute mean individual fan airflow rate based on verified fan operational status, dP and/or the degraded fan curves.
- 9.5.2. The equation to standardize the actual airflow (m³/s) to the moist standard airflow (sm³/s) is as follows:

$$Q' = \frac{Q \cdot P \cdot (273.15 + T')}{P' \cdot (273.15 + T)} \quad (4)$$

Where:

- Q' = Moist standard airflow (sm³/s)
- P' = Standard pressure (1 atm)
- T' = Standard temperature (20°C)
- Q = Outlet airflow rate at T (m³/s)
- P = Pressure at sampling location (atm)
- T = Temperature at sampling location (°C)

- 9.5.3. Calculate the dry-standard airflow rate (Q'') by excluding moisture. The equation to standardize the moist standard airflow (m³/s) to dry standard airflow (dsm³/s) is as follows:

$$Q'' = (1 - W) \cdot Q' \quad (5)$$

Where:

- Q'' = Dry standard airflow (dsm³/s)
- W = Humidity ratio (See Appendix E of SOP B4)

- 9.5.4. Assign zero airflow for any non-functional fans.
- 9.5.5. Sum the individual fan airflow rates to obtain the barn ventilation rate.

10. Data and Records Management

- 10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet that is designated for this method. Supplement this electronic record with a bound record book designated for the method, which should also contain bound copies of the electronic record.
- 10.2. Manage all data according to SOP B5.
- 10.3. Document all data and information on field data sheets, and site logbooks with permanent ink, or in electronic field notes. Overstrike errors in writing with a single line. Initial and date all such corrections.

11. Quality Control and Quality Assurance

- 11.1. Utilize all data, including fan stage operation, anemometer, vibration sensor, observation, site field-notes, and any maintenance note provided by the farm to ensure quality control of individual fan airflow rate. Exclude fans that were idle from the pool of working fans for total ventilation rate calculation.
- 11.2. Conduct weekly verification of fan stage operation monitoring, anemometer, rpm sensor, current switch and/or vibration sensor operations and verify that the operational status signals agree with individual fan operation. Verify that the individual fans are operating when the ventilation stage to which they belong is operating.
- 11.3. Perform FANS unit calibration at the University of Illinois BESS Lab annually to ensure that the unit is functioning properly.
- 11.4. Conduct FANS test of each fan at least annually. After each FANS test, verify the degraded fan curves and/or degradation correction factors determined using the previous FANS test data, and correct for elapsed time if deemed necessary.
- 11.5. If possible, perform new FANS test on any individual fan that has undergone maintenance that is likely to significantly change its performance (such as installation of a new shutter, belt, motor, etc).

12. References

- 12.1. Gates, R.S., J.D. Simmons, K.D. Casey, T.J. Greis, H. Xin, E.F. Wheeler, C.L. King, and J.R. Barnett. 2002. Fan Assessment Numeration System (FANS) design and calibration specifications. ASAE Paper No. 024124. St. Joseph, MI: American Society of Agricultural Engineers.
- 12.2. Heber, A.J., J.-Q. Ni, and T.T. Lim. 2003. Air quality measurements at a laying hen house: Experimental methods. International Symposium on Control of Gaseous and Odour Emissions from Animal Production Facilities, Horsens, Denmark, June 2-4
- 12.3. Lim, T.T., A.J. Heber, and J. Ni. 2003. Air quality measurement at a laying hen house: Ventilation airflow measurement. ASAE Meeting Paper No. 034079. St. Joseph, MI: American Society of Agricultural Engineers.

- 12.4. Ni, J.-Q., A.J. Heber, T.T. Lim, and C.A. Diehl. 2005. A low-cost technique to monitor ventilation fan operations in animal buildings to increase air emission measurement quality. Seventh International Livestock Environment Symposium, Beijing, China.
- 12.5. SOP A1. 2006. Laboratory Testing of Ventilation Fan Performance. Standard Operating Procedure A1, v. 0.0. Purdue Ag Air Quality Lab.
- 12.6. SOP A2. 2007. Measurement of Ventilation Fan Flowrate Using the Fan Assessment Numeration System (FANS). Standard Operating Procedure A2, v. 1.0. Purdue Ag Air Quality Lab.
- 12.7. SOP A3. 2007. Ventilation Fan Air Velocity Measurements Using Open Impeller Anemometry. Standard Operating Procedure A3, v. 1.0. Purdue Ag Air Quality Lab.
- 12.8. SOP A5. 2007. Measurement of Fan Static Pressure in Livestock Buildings with Setra Model 267 Differential Pressure Transducer. Standard Operating Procedure A5, v. 1.0. Purdue Ag Air Quality Lab.
- 12.9. SOP A8. 2006. Ventilation Fan Airflow Measurements using the Air Velocity Traverse Method. Standard Operating Procedure A8, v. 0.0. Purdue Ag Air Quality Lab.
- 12.10. SOP B2. 2007. Data Acquisition and Control Software (AirDAC). Standard Operating Procedure B2, v. 1.0. Purdue Ag Air Quality Lab.
- 12.11. SOP B3. 2006. Air Data Pre-Processing Software. Standard Operating Procedure B3, v. 0.0. Purdue Ag Air Quality Lab.
- 12.12. SOP B4. 2008. Calculation and Reporting of Air Emissions from Barns. Standard Operating Procedure B4, v. 1.0. Purdue Ag Air Quality Lab
- 12.13. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5, v. 0.0. Purdue Ag Air Quality Lab.
- 12.14. SOP B6. 2007. Data Processing Software (CAPECAB). Standard Operating Procedure B6, v. 1.0. Purdue Ag Air Quality Lab.
- 12.15. SOP S1. 2006. Producer Collaborations at Barn Monitoring Sites. Standard Operating Procedure S1, v. 0.0. Purdue Ag Air Quality Lab.
- 12.16. SOP S4. 2006. Process Relay Monitoring. Standard Operating Procedure S4, v. 0.0. Purdue Ag Air Quality Lab.

**MEASUREMENT OF FAN STATIC PRESSURE IN LIVESTOCK
BUILDINGS WITH SETRA MODEL 260 DIFFERENTIAL PRESSURE
TRANSDUCER**

Standard Operating Procure (SOP) A5

**MEASUREMENT OF FAN STATIC PRESSURE IN LIVESTOCK BUILDINGS WITH
SETRA MODEL 260 DIFFERENTIAL PRESSURE TRANSDUCER
Standard Operating Procedure (SOP) A5**

Prepared by

Teng-Teoh Lim, Bill W. Bogan, and Erin L. Cortus

Reviewed by

Steve J. Hoff (Iowa State Univ.), Richard Gates (Univ. of Kentucky), and Albert J. Heber

Effective Date: November 13, 2007

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions of Terms and Acronyms	3
4. Health and Safety	4
5. Cautions.....	4
6. Interferences	4
7. Personnel Qualifications	4
8. Equipment and Supplies	5
9. Procedures.....	5
10. Data and Records Management	8
11. Quality Control and Quality Assurance.....	8
12. References	9
13. Contact Information.....	9

1. Scope and Applicability

- 1.1. The Setra Model 260 multi-range pressure transducer is useful for measuring building differential pressure (ΔP) in facilities such as livestock buildings with negative pressure ventilation systems. Estimating air flow rate of ventilation fans based on actual fan performance curves requires measurement of ΔP .
- 1.2. The Setra Model 260 MS3 has four unidirectional (25, 50, 100 and 250 Pa FS) and four bidirectional ranges (± 25 , 50, 100 and 250 Pa FS) that can be set in the field. A bidirectional range of 100 Pa is the default range for NAEMS measurements.
- 1.3. The transducer measures the ΔP and converts the readings to a proportional electrical output. The manufacturer's stated transducer accuracy is $\pm 1\%$ full scale at constant temperature. The operating temperature limits are 0 to 50°C (32 to 122°F).
- 1.4. The negative static pressure in a barn is created by the exhaust ventilation fans and is typically -60 Pa to +10 Pa. High winds are able to pressurize a barn even with exhaust fans running. Therefore the transducer is bidirectional with a scale of -100 to +100 Pa.
- 1.5. The method also applies to monitoring the ΔP in the instrument shelter created by the heating, ventilating and air conditioning system, which is usually -10 Pa to +10 Pa.

2. Summary of Method

This method continuously monitors the static pressure of barn exhaust fans, for computation of building airflow rate. The Setra Model 260 transducer senses ΔP between two points with a sensitive electrical capacitor. The transducer consists of two closely-spaced, parallel, electrically-isolated metallic surfaces, one of which is essentially a diaphragm capable of slight flexing under positive or negative pressure. The diaphragm is constructed of a low-hysteresis material or a proprietary compound of fused glass and ceramic (Setraceram™). These firmly secured surfaces (or plates) are mounted such that a slight mechanical flexing of the assembly, caused by a minor change in applied pressure, alters the gap between them (creating, in effect, a variable capacitor). The resulting change in capacitance is detected and converted to a proportional analog signal. In this method, the positive (high) pressure port senses the pressure inside the building, while the reference (low) pressure port is in contact with the outside atmosphere. Polyethylene (PE) tubing is used to connect the pressure port in the barn or other locations. Typically, for facilities with fans on more than one sidewall, the ΔP across each wall with fans will be measured to account for wind interference.

3. Definitions of Terms and Acronyms

- | | | |
|------|-------------------|---|
| 3.1. | DAC | Data acquisition and control |
| 3.2. | ΔP | Differential static pressure |
| 3.3. | PAAQL | Purdue Agricultural Air Quality Laboratory |
| 3.4. | PE | Polyethylene |
| 3.5. | QAPP | Quality Assurance Project Plan |
| 3.6. | SOP | Standard Operating Procedure |
| 3.7. | Static Pressure | Pressure that exists in the absence of air velocity |
| 3.8. | Velocity Pressure | Pressure associated with or caused by air movement |

4. Health and Safety

- 4.1. Be careful when working with the electrical power connections.

5. Cautions

- 5.1. Minimize exposure to liquids or corrosive gases, which can damage the pressure transducer unit. If possible, place the sensor in a protected location, and run tubes to remote pressure ports in the barn.
- 5.2. Install a pressure snubber orifice at the end of a polyethylene (PE) tube to minimize effects of air movement (from wind, fans, inlets, etc.) on the measurement. Installing the orifice will help reduce pressure pulsations and sudden fluctuations. The ports used for indoor measurements will be located far away from exhaust fans to minimize the effect of air speed (velocity pressure), see section 8.
- 5.3. Protect the end of the open PE tube with material such as foam to keep dust and insects out of the tube, unless a pressure snubber or orifice is used.
- 5.4. Keep the end of the PE tube which is used to measure ambient pressure pointing vertically downward, to prevent moisture from becoming trapped inside the tube. Installing a membrane filter holder at the end of ambient tube has proven effective in previous measurement tests.
- 5.5. Do not adjust the factory calibration settings of the transducer unless the transducer goes out of calibration (see Section 9.3.5).
- 5.6. Pressure outside the limits of the transducer can damage it.
- 5.7. Avoid running the PE tubing through cold areas, especially near the pressure port, to prevent condensation of water vapor that migrates into the tube by diffusion.

6. Interferences

- 6.1. Use of other than the recommended tube sizes (See Step 9.1.2) may lead to slower-than-optimal response times.
- 6.2. Airflow and air circulation, with streamlines directed at pressure ports, caused by wind and nearby operating fans may create pressure pulsations and surges due to velocity pressure. Minimize the interference by following these procedures:
 - 6.2.1. Measure the static pressure differential across each sidewall that has exhaust fans because static pressure varies between the four walls of a barn due to wind.
 - 6.2.2. Position the static pressure probe or tap perpendicular to the streamlines of flow.
 - 6.2.3. Use small orifices or pressure snubbers in the static pressure ports.

7. Personnel Qualifications

- 7.1. Personnel should be trained in the use of the instrument before initiating the procedures listed in this SOP.
- 7.2. Each analyst must read the entire instrument manual and this SOP before operating or calibrating the instrument.

8. Equipment and Supplies

- 8.1. Setra Model 260 MS3 Differential Pressure Transducer (Setra Systems, Boxborough, MA)
- 8.2. Polyethylene tube for connecting unit ports to the pressure measuring locations (See Step 9.1.2)
- 8.3. Pressure snubber or screened orifice (Model BWFS64, O'Keefe Controls Co., Trumbull, CT) for end of tube, used for indoor measurement.
- 8.4. Air conditioner filter material (or equivalent) to fit inside the end of the tube and prevent dust and insects from entering tube.
- 8.5. Filter holder with 1/4" inlet/outlet ports (Model 4-47-4, Savillex Corporation, Minnetonka, MN, or equivalent) for end of tube, used for ambient measurement
- 8.6. NEMA 4 rated or equivalent junction box (for sensors located in the barn only)

9. Procedures

- 9.1. Installation
 - 9.1.1. Mount the transducer in the vertical position inside the OFIS or inside an appropriate protective enclosure in the barn. The transducers are factory calibrated in the vertical position.
 - 9.1.2. Connect PE tube between the pressure port at the inside or outside measurement location and the pressure port of the transducer located inside a protective shelter (eg., on-farm instrument shelter, OFIS). The selection of tube size depends on its length. To ensure the shortest response time, the manual recommends the following inside diameters.
 - 9.1.2.1. 0-100 ft: 3/16 in
 - 9.1.2.2. 100-300 ft: 1/4 in
 - 9.1.2.3. 300-900 ft: 3/8 in
 - 9.1.3. Connect the atmospheric (outside) static pressure tube to the reference (low) pressure port, which is located on the bottom of the unit (Labeled "LOW").
 - 9.1.4. Connect the inside static pressure tube to the positive (high) pressure port, which is located on the bottom of the unit (Labeled "HIGH"). If the inside static pressure is less than atmospheric, the reading will be negative.
 - 9.1.5. Protect the end of the pressure tube with foam to keep dust and insects out.
 - 9.1.6. Pressure snubbers or screened orifices can be used at each sampling point to minimize effects of air movement (wind) on the pressure measurement.
- 9.2. Electrical Connections
 - 9.2.1. The voltage output is a 3-wire circuit, with three terminals for wiring (Fig. 1). The terminals are clearly identified on the circuit board inside the transducer. The output signal range is 0-5 VDC for excitation of 10.2 to 42 VDC, and 0-10 VDC for excitation of 13 to 42 VDC. Because the sensor is located in the OFIS, 4-20 mA is not needed to avoid noise due to long signal wires.
 - 9.2.2. Connect the appropriate power and ground cables to the terminals.
 - 9.2.3. Connect the analog signal (VDC) output of the analyzer to terminals on the data acquisition system.

- 9.2.4. Use the slide switches to set the pressure range, mode (uni- or bi-directional) and output range (Fig. 2).
- 9.2.5. Enter the analyzer's analog signal and measurement ranges into the AirDAC software so that ΔP readings can be converted, displayed, and recorded.
- 9.2.6. Turn on the power to the transducer.

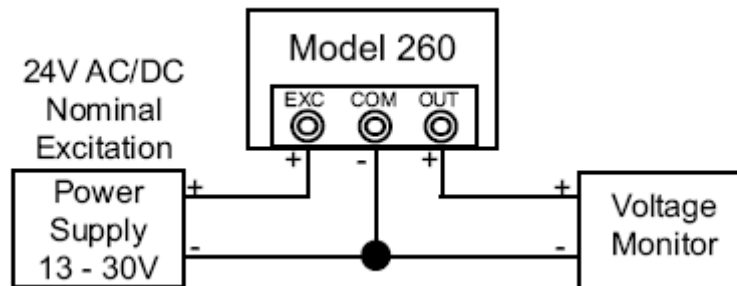


Figure 1. Model 260 pressure transducer electrical connection terminals (Setra Systems, 2007).

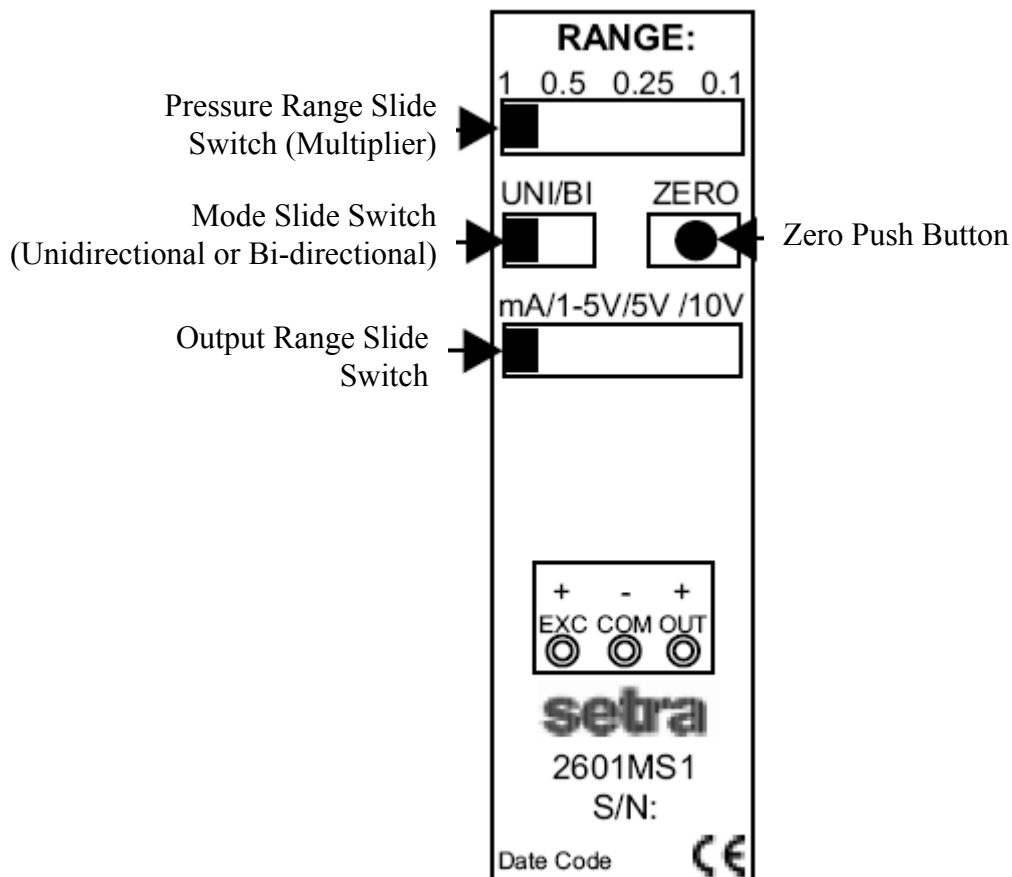


Figure 2. Model 260 pressure transducer compartment containing slide switches, zero push button and electrical connection points (Setra Systems, 2007).

9.3. Calibration

- 9.3.1. To apply zero pressure, disconnect the PE tubes of both pressure ports and shunt the ports together.
- 9.3.1.1. A short piece of tube can be used to test the sensor itself.
- 9.3.1.2. A long tube can be used to connect the ends of the two pressure tubes in the barn. This method is used to check the entire pressure sampling system.
- 9.3.1.3. Press the zero push button (Fig. 2) to adjust the zero offset.
- 9.3.2. Apply nonzero pressure simultaneously to transducer and a reference transducer or manometer, or use a NIST-traceable device to apply a reference pressure. Instructions for simultaneous pressure measurements by two devices are provided.
- 9.3.2.1. Disconnect tubes from low ports of both transducers and connect them, in parallel, using a length of PE tubing.
- 9.3.2.2. Connect the high ports of both transducers in parallel using a PE “T” or “Y” connector, with a stable pressure applied to connector’s third port (Fig. 2).
- 9.3.2.2.1. Short tubes can be used to test the sensor itself.
- 9.3.2.2.2. The preferred method is to apply the stable pressure at each pressure monitoring point with the “T” or “Y” connector near the transducers (Fig. 2).
- 9.3.2.3. Application of reference pressure
- 9.3.2.3.1. Positive: Place weights on top of partially filled single-port bag.
- 9.3.2.3.2. Negative: Monitor negative pressure created by barn exhaust fans.
- 9.3.2.3.3. Both: A syringe that can be drawn or pushed into the connector, as shown in Fig. 2.

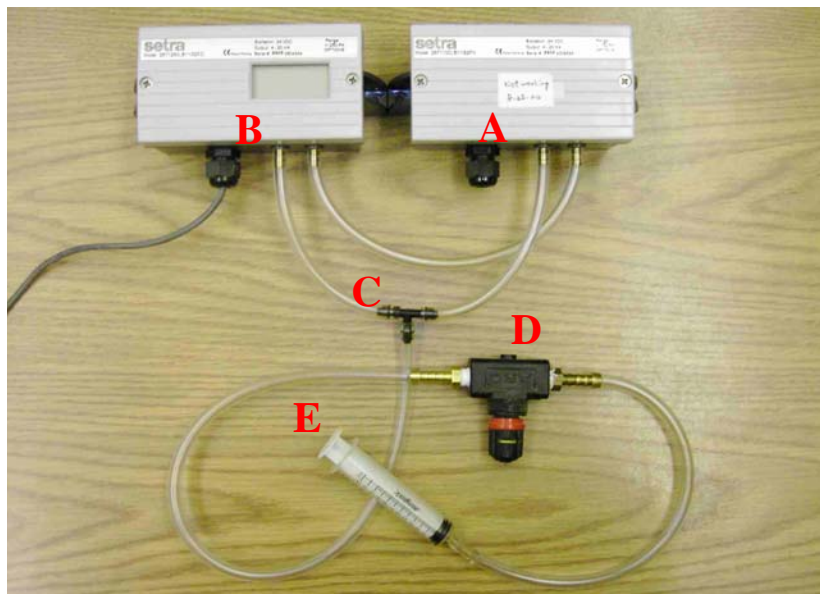


Figure 2. Calibration of a pressure transducer (A) with a calibrated reference unit (B), connected in parallel using a “T” connector (C). A pressure valve (D) and syringe (E) were used to create stable high or low pressure. Part B can be replaced with a micromanometer, in accordance with Section 9.3.2.4.1. Parts D and E can be replaced by a precision pressure generator and calibration unit (e.g. Furness Company).

- 9.3.2.4. Measurement of reference pressure
 - 9.3.2.4.1. Precision micromanometer with resolution of ± 0.001 in. of water
 - 9.3.2.4.2. Identical or more accurate Setra transducer that is calibrated and verified with a precision check upon receipt.
 - 9.3.2.4.3. Inclined manometer
- 9.3.3. All Model 260 pressure transducers are factory-calibrated using NIST-traceable standards, and should require no further adjustment upon receipt. However, conduct a precision check upon receipt and before collecting data to verify transducer integrity.
- 9.3.4. Conduct zero and/or span offsets based on the precision checks. The correction can be made through adjustments to the data acquisition and control (DAC, SOP B2) system, or in the data processing (SOP B6).
- 9.3.5. Adjust sensor only if error is more than 5% of full scale, e.g. ± 5 Pa for ± 100 Pa sensor. Adjusting the sensor after factory calibration voids the certification provided by the manufacturer.
 - 9.3.5.1. Zero and span adjustments will only be made when using a NIST-traceable pressure standard with at least the same accuracy as the Setra transducer ($< \pm 1\%$ FS),
 - 9.3.5.2. Record the reference pressure and the as-found value of the transducer.
- 9.4. Computer hardware & software
 - 9.4.1. AirDAC (SOP B2)
 - 9.4.2. ADPP (SOP B3)
 - 9.4.3. CAPECAB (SOP B6)

10. Data and Records Management

- 10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which will contain copies of the electronic record.
- 10.2. Manage all data according to SOP B5.
- 10.3. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes.

11. Quality Control and Quality Assurance

- 11.1. Conduct zero checks at least monthly or according to the QAPP.
- 11.2. Conduct span checks before and after data collection, and semiannually, or according to the QAPP.
- 11.3. Attention will be given to visually checking measurement data frequently. Sudden variations may indicate that tube has been kinked, squeezed by a heavy object, or clogged. Conduct correction action if this occurs.
- 11.4. Select proper size (ID) and thickness of the PE tube (See Step 9.1.2) to ensure quick response time.
- 11.5. Since all Model 260 pressure transducers are factory-calibrated using NIST-traceable standards, avoid adjustment if drift is less than 5% of full scale. If is preferable to correct for zero and/or span offsets during post processing collected data.

- 11.6. For barns with fans on more than one sidewall, the differential static pressure across each sidewall will be measured. A common static pressure port is used inside the building and the difference in pressure between the building pressure and each sidewall is measured. Thus, one static pressure sensor is required for each sidewall.

12. References

- 12.1. Setra Systems. 2007. Setra Pressure Measurement Division - Technology. Setra Systems, Inc., <http://www.setra.com/tra/tec/tec.htm>. Accessed 11/9/07.
- 12.2. Setra Systems. 2007. Installation Guide - "Multi-Sense" Model 260 Series Multi-Range Differential Pressure Transducer. Setra Systems, Boxborough, MA.
- 12.3. AMCA Standard 210-85. 1985. Laboratory methods of testing fans for rating. Air Moving and Conditioning Association, Inc., Arlington Heights, IL.
- 12.4. SOP B2. 2006. Data Acquisition and Control Software (AirDAC). Standard Operating Procedure B2. Purdue Ag Air Quality Lab.
- 12.5. SOP B3. 2006. AirDAC Raw Data Inspection System (ARDI). Standard Operating Procedure B3. Purdue Ag Air Quality Lab.
- 12.6. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 12.7. SOP B6. 2006. Data Processing Software (CAPECAB). Standard Operating Procedure B6. Purdue Ag Air Quality Lab.

13. Contact Information

- 13.1. Manufacturer:
SETRA, Inc.
159 Swanson Road
Boxborough, MA 01719
1-800-257-3872
<http://www.setra.com/contact.htm>
- 13.2. PAAQL: odor@purdue.edu, heber@purdue.edu, <http://www.AgAirQuality.com>.

**AIR VELOCITY MEASUREMENTS WITH THE RM YOUNG
MODEL 81000 3-DIMENSIONAL ULTRASONIC ANEMOMETER
Standard Operating Procedure (SOP) A6**

**AIR VELOCITY MEASUREMENTS WITH THE RM YOUNG
MODEL 81000 3-DIMENSIONAL ULTRASONIC ANEMOMETER
Standard Operating Procedure (SOP) A6**

Prepared by

Richard H. Grant, Juan Carlos Ramirez, and Bill W. Bogan

Reviewed by

Ji-Qin Ni and Albert J. Heber

Effective Date: November 6, 2007

**PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)
Agricultural and Biological Engineering, Purdue University
West Lafayette, IN 47907**

Table of Contents

	Page
1. Scope and Applicability.....	3
2. Summary of Method.....	3
3. Definitions.....	4
4. Health and Safety.....	4
5. Cautions.....	4
6. Interferences.....	4
7. Personnel Qualifications.....	5
8. Equipment and Supplies.....	5
9. Procedures.....	5
10. Data and Records Management.....	10
11. Quality Control and Quality Assurance.....	10
12. References.....	11
13. Contact Information.....	12

1. Scope and Applicability

- 1.1. Measurements of air velocity through a livestock barn are necessary for determining the ventilation rate, which is, in turn, necessary for estimating emissions of airborne contaminants from the barn.
- 1.2. This SOP describes procedures for continuously monitoring the velocity of air moving in and out of an animal barn (for example, an open dairy barn), using multiple ultrasonic anemometers.
- 1.3. This procedure applies only to the RM Young Model 81000 3-D ultrasonic anemometer.
 - 1.3.1. The range of air velocities that can be measured is 0.01 to 40 m/s.

2. Summary of Method

The RM Young Model 81000 Ultrasonic 3-D Anemometer (Fig. 1) measures three-dimensional air velocity by determining the transit times of ultrasonic acoustic signals. Anemometers can be installed at any opening or point inside a barn where airflow measurements are needed, such as the ventilation openings of naturally-ventilated barns, which are usually in the side walls, the end walls, and the roof ridge (Fig. 2). Other places where anemometers could be used include throughways and connecting alleys within a group of barns. Each anemometer monitors the air speed through a section of an opening or passageway. The North direction of the anemometer is positioned to face the inside of the barn, and is therefore perpendicular to the plane of the barn opening that the anemometer is monitoring. Measurement data from each anemometer are available as four, 0 – 5000 mV analog voltage outputs, corresponding to air speed in the x, y, and z directions (U, V, and W) and sonic temperature. The analog outputs are connected to the data acquisition (DAQ) system for real-time monitoring, and the data are recorded in the DAQ computer. Subsequent data processing converts the measured air velocity components to the air speed perpendicular to the barn opening surface. Air flow rate in and out of the barn through that opening or passageway is obtained by multiplying the air speed perpendicular to the opening surface with the area of that opening.

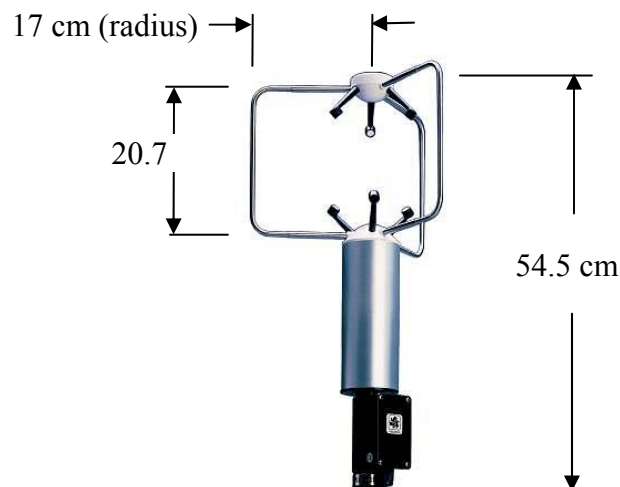


Figure 1. R.M. Young Model 81000 Ultrasonic 3-D Anemometer (Campbell Scientific Co., 2006).

3. Definitions

- 3.1. ADPP Air Data Pre-Processing software, written in Visual Basic for Applications in MS Excel
- 3.2. AirDAC Air quality monitoring Data Acquisition and Control program written in LabVIEW (SOP B2).
- 3.3. Azimuth Horizontal direction of airflow when the 3-D ultrasonic anemometer is used to measure wind velocity, degrees. It is calculated in the x-y plane.
- 3.4. DAQ Data acquisition
- 3.5. Elevation Vertical angle of airflow when the 3-D ultrasonic anemometer is used to measure wind velocity, degrees. It is the angle between the z component and the x-y resultant.
- 3.6. OFIS On-farm instrument shelter
- 3.7. PAAQL Purdue Agricultural Air Quality Laboratory
- 3.8. QAPP Quality Assurance Project Plan
- 3.9. U, V, W Air velocity components in the x, y, and z directions, respectively
- 3.10. 3-D Three-dimensional

4. Health and Safety

- 4.1. When installing or inspecting the anemometers at the roof ridge opening of a barn, follow the appropriate safety procedures for working on rooftops and with ladders. These are listed in SOP E5.

5. Cautions

- 5.1. The anemometer has electrical components, and proper care should be used as with all electrical equipment.
- 5.2. Handle the anemometer carefully to avoid physical damage.
- 5.3. Turn on the power to the anemometer only after all connections are made and verified.
- 5.4. If the anemometer is mounted in any orientation other than vertical (e.g. horizontal or upside-down), seal the openings around the cable entering the unit with electrical tape or silicone caulk to keep water from damaging the electronics.
- 5.5. Be sure that the anemometer is earth-grounded (Refer to page 8 of the instrument manual, RM Young Company, 2005).

6. Interferences

- 6.1. Incorrect siting will cause inaccuracies in the measurements due to failure of assumptions of spatial homogeneity and temporal stationarity of the air velocity field.
- 6.2. Anemometer response under light mist or heavy fog can be diminished, due to accumulation of water droplets on the anemometer transducers.
- 6.3. The anemometer does not respond under conditions in which freezing rain, heavy frost and/or snow accumulates on the transducers.

- 6.4. Accumulated dust, dirt, or fly waste on the transducers can also affect performance. Inspect the unit periodically, and clean the transducer surfaces if necessary.
- 6.5. The particular configuration of this anemometer can encourage nesting by birds.
- 6.6. Wake flow around support struts reduces anemometer performance.
- 6.7. If the anemometer is mounted vertically, a wind angle of attack (relative to the horizontal) of $>60^\circ$ will cause less accurate readings due to the physical structure of the sensor. The magnitude of these inaccuracies is unknown. Such angles of attack are introduced only when the anemometer is oriented nonperpendicular to the air velocity being measured.
- 6.8. A 1° tilt of anemometer induces an error in the measurement of approximately 6% in the correlations of air velocity deviations and 3% in the derived friction velocity with tilt.
 - 6.8.1. Errors caused by slight ($<1^\circ$) tilt of the anemometer can be corrected mathematically.
 - 6.8.2. However, if the anemometer is tilted $>1^\circ$ the readings will be non-linear and cannot be corrected.

7. Personnel Qualifications

- 7.1. Personnel must read and understand the instruction manual (RM Young Company, 2005), this SOP, and SOPs B1 and B2 before installing and operating this anemometer.

8. Equipment and Supplies

- 8.1. R.M. Young Model 81000 3-D Ultrasonic Anemometer (R.M. Young, Traverse City, MI) with manual
- 8.2. 12 – 24 VDC power supply (110 mA for each anemometer)
- 8.3. Three (if sonic temperature data are not acquired) or four (if sonic temperature is included) different DAQ analog input channels for each anemometer
- 8.4. RS232 cable
- 8.5. Anemometer power and signal cable (8-conductor, 22-gage, shielded)
- 8.6. 1.34-in. (OD) diameter mounting post or pipe (to hold the instrument)
- 8.7. Clamps or other hardware to attach mounting posts to the supporting structure
- 8.8. Multimeter
- 8.9. Precision level
- 8.10. Compass
- 8.11. #2 common head screwdriver
- 8.12. 1/8" slotted screwdriver
- 8.13. Computer
- 8.14. AirDAC program (SOP B2)
- 8.15. DAQ board with hardware (SOP B1)

9. Procedures

- 9.1. Anemometer installation
 - 9.1.1. The factory default configurations (Section 3.0 of the manual) are 3D wind speed, wind direction, azimuth, and sonic temperature. To change to U, V, W, and sonic

temperature, follow the procedures described in Sections 4.0, 6.0 and 6.7.1 in the instrument manual, as follows:

- 9.1.1.1. Remove junction box cover. Connect power and signal wires to terminals as indicated in wiring diagram (on p. 6 of the manual) for RS-232 output. Connect the RS232 cable to the COM port of a laptop or PC.
- 9.1.1.2. Start HyperTerm or an equivalent serial communications program with a baud rate of 38400, and flow control set to NONE.
- 9.1.1.3. While the anemometer is powered and operating, send the ESC character (ASCII 27) the time in rapid succession. This will access the anemometer's COMMAND menu.
- 9.1.1.4. Send "S" to access SETUP mode.
- 9.1.1.5. From the SETUP menu, send "S" for SER OUTPUT FORMAT.
- 9.1.1.6. From the SERIAL OUTPUT FORMAT menu, send "1" to access the CUSTOM format.
- 9.1.1.7. Send "5B" to setup output of U, V, W, and sonic temperature (Ts).
- 9.1.1.8. Send "X" to return to main (COMMAND) menu, and "X" again to return to "OPERATE" mode.
- 9.1.2. Establish the exact locations where the anemometers should be mounted according to the Site Monitoring Plan. Some locations may require more than one anemometer, while a single one may suffice for other locations. In general:
 - 9.1.2.1. Measuring airflow through the ridge opening along the barn length will require multiple anemometers.
 - 9.1.2.2. Measuring airflow through the sidewall opening along the barn length will require multiple anemometers.
 - 9.1.2.3. One or more anemometers may be required to measure airflow through the face of the opening at the end walls (short side) of the barn, depending on its configuration. This end side could have a door (to allow the entrance of the feeding truck) or be totally open (without wall).
 - 9.1.2.4. Measuring airflow through a connecting passageway may only require one anemometer.
 - 9.1.2.5. In all of the above cases, the anemometer should generally be located in the plane of the narrowest part of the opening. For example, on the side opening of a naturally ventilated dairy barn, the curtains are adjusted vertically, leaving a narrow opening between the eave and the curtain in winter. The 3-D anemometer should be placed in the center of this narrow opening.
 - 9.1.2.6. To define the best vertical location of the anemometers when the sidewalls have no restrictions (for example, no curtains or walls), locate two to four anemometers (depending of the opening height) vertically in the horizontal center of the sidewall. Divide the vertical dimension of the opening into equal lengths, and locate the anemometers at the center of each length. Compute the average air velocity and select the location where the air velocity is closest to the average to locate all the anemometers on that side.
- 9.1.3. Mount each anemometer securely on the mounting post, such that it will not twist, rotate, or sway. Any movement will compromise the measurements.

- 9.1.4. Mount the anemometers with the junction box of the anemometer facing outside the barn, so that the North (the strut opposite the junction box) points into the barn and is perpendicular to the building structure opening that the anemometer is monitoring (Fig. 3). Refer to page 9 in the instrument manual (RM Young Company, 2005) for further information regarding anemometer orientation.
- 9.1.5. Orient the anemometer mounting arm in the position that best facilitates easy installation, while keeping the North oriented correctly (Section 9.1.4).
- 9.1.6. Connect the anemometer earth ground terminal to a suitable earth ground.
- 9.2. Analog signal and power connection and DAQ settings
 - 9.2.1. Consult the QAPP to determine if sonic temperature will be measured or not. This will determine the number of DAQ channels needed (Section 8.3)
 - 9.2.2. Remove the junction box cover. Connect the multi-conductor power and signal cable to terminals as indicated in the wiring diagram on page 8 of the instrument manual (RM Young Company, 2005). Connect the other end of the cable to the data acquisition hardware (SOP B1), and to the anemometer power supply.
 - 9.2.3. Verify that all connections are correct before turning on the DAQ system power supply.
 - 9.2.4. Turn on the power to the DAQ system.
 - 9.2.5. Start AirDAC (SOP B2). Go to the “DAC” tab, and configure the anemometer signal according to Table 2. Apply and save the configuration.
 - 9.2.6. Check the real-time data displayed in rows 15 to 17 in the DAC tab or in the history plots (SOP B2).

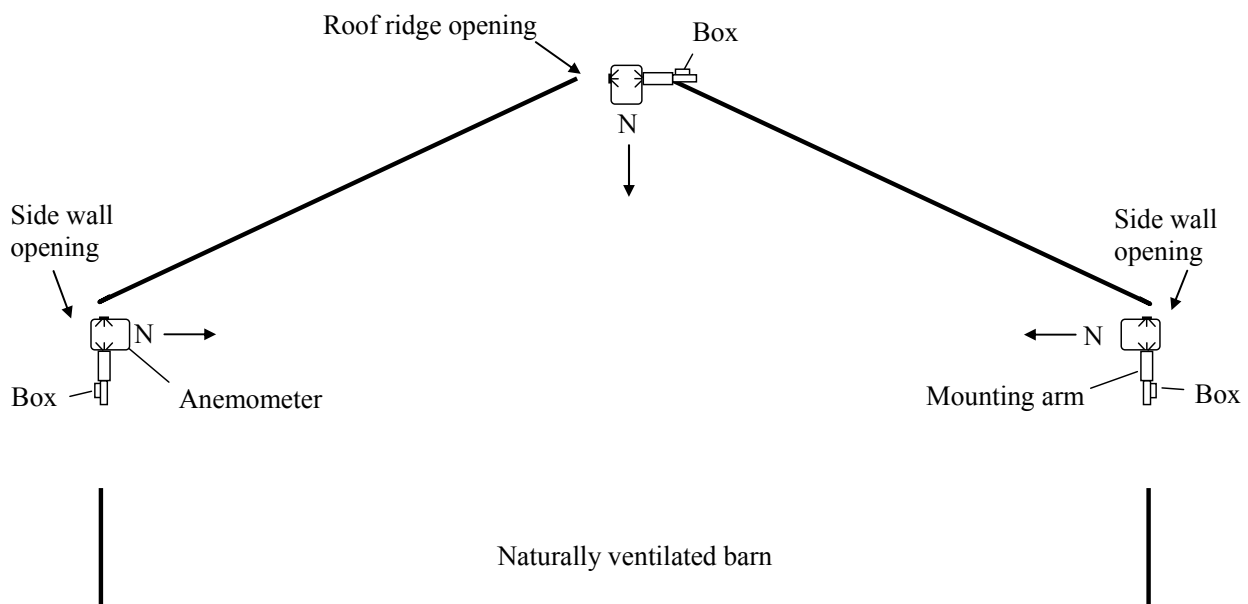


Figure 2. Orientation of 3-D anemometers in wall openings and roof ridge openings.

- 9.2.6.1. If the anemometer has analog output signals, verify that the DAQ hardware and software are working properly.

- 9.2.7. If the signals acquired by the data acquisition system are not valid (outside of the range presented in Table 1):
 - 9.2.7.1. Check for obstructions to the acoustic path of the anemometer. Invalid measurements can occur when the acoustic path of the sonic signal is blocked or internal circuits fail. Acoustic blockage may be caused by rain drops, ice, snow, or other debris.
 - 9.2.7.2. Check the DAQ hardware and software configuration.
 - 9.2.7.3. If the acoustic path is clear and the DAQ system is normal, but the anemometer is still not working properly, return the anemometer to the manufacturer for repair or replacement.
- 9.3. Initial checks and periodic verification
 - 9.3.1. Follow the detailed descriptions in Section 3.0 (“Initial Checkout”) of the instrument manual (RM Young Company, 2005) to conduct the initial check. These checks can either be done in the trailer (with the anemometer connected to the DAQ system), or after installation.
 - 9.3.1.1. Verify that the zero reading is stable by enclosing the unit in a bag or box. Replace the original packaging foam over the anemometer, and slide its shipping bag over it to enclose the unit.
 - 9.3.1.1.1. Record the anemometer biases in the U, V, and W components.
 - 9.3.1.1.2. If the anemometer does not indicate values of less than 0.1 m/s in all three components, send it back to the manufacturer for repair or replacement.
 - 9.3.1.2. Verify sensor response by gently blowing through the measuring section of the anemometer and observing the output to the DAQ system. Do this from two horizontal directions (i.e. x and y).
 - 9.3.2. If sonic temperature is being measured, check the temperature reading of each installed 3-D ultrasonic anemometer annually, or as stated in the QAPP, by comparing it (in place) with a NIST-traceable reference thermometer or other NIST-traceable standard.
 - 9.3.2.1. If the anemometer agrees to within 2°C, leave it in place. If the anemometer reads $>\pm 2^\circ\text{C}$ different from the reference thermometer, replace the signal wire and repeat the test. If the reading is still $>2^\circ\text{C}$ different from the reference thermometer, replace the 3-D ultrasonic anemometer and repeat the test. Send the old anemometer to the manufacturer for repair or maintenance.
 - 9.3.3. Repeat the zero check described above (Section 9.3.1.1) at the frequency specified by the QAPP.
- 9.4. Troubleshooting
 - 9.4.1. If no signals are shown in the AirDAC for the anemometer:
 - 9.4.1.1. Verify that the anemometer is powered by 12-24 VDC. Open the junction box of the anemometer and measure the voltage between +PWR and PWR REF with a multimeter. If there is no voltage, check the continuity of wires and check the power supply inside the OFIS.
 - 9.4.1.2. If the anemometer is properly powered, check the analog output voltage at terminals V1, V2, V3, and V4 relative to VREF. The voltages should be between 0 and 5000 mV. If no analog output is detected, return the anemometer to the manufacturer for repair or replacement.

Table 1. Normal value range for each of the parameters measured.

Parameter	Specification
Wind Speed (U,V,W)	0 to 40 m/s (0 to 90 mph)
Sonic temperature	-50°C to 50°C

Table 2. Proper configuration of anemometer analog signals in AirDAC for U,V,W and sonic temperature.

1. Data heading	A#n U, m/s	A#n V, m/s	A#n W, m/s	A#n T, °C
2. Digit of precision	2	2	2	1
3. Signal lo (SL)	0	0	0	0
4. Signal hi (SH)	5	5	5	5
5. Range lo (RL)	0	0	0	220
6. Range hi (RH)	50	50	50	320
7. Absolute (abs)	0	0	0	0
8. Slope (A)	1	1	1	1
9. Intercept (B)	0	0	0	-273
10. Global Ch#				
11. Limit: Min	0	0	0	-50
12. Limit: Max	15	15	15	40
13. Hysteresis	2	2	2	5
14. Email { ,DO#}	1	1	1	1

Note: A#n, anemometer number (n = 1, 2, ...); T, sonic temperature. Fill in “10. Global Ch#” according to the actual DAQ hardware configuration. “11. Limit: Min” to “14. Email { ,DO#}” are for real-time data monitoring (Refer to SOP B2 for details).

- 9.4.1.3. If the anemometer has analog output signals, verify that the DAQ hardware and software are working properly.
- 9.4.2. If the signals acquired by the data acquisition system are not valid:
 - 9.4.2.1. Check for obstructions to the acoustic path of the anemometer. Invalid measurements can occur when the acoustic path of the sonic signal is blocked or internal circuits fail. Acoustic blockage may be caused by rain drops, ice, snow, or other debris.
 - 9.4.2.2. Check the DAQ hardware and software configuration.
 - 9.4.2.3. If the acoustic path is clear and the DAQ system is normal, but the anemometer is still not working properly, return the anemometer to the manufacturer for repair or replacement.

10. Data and Records Management

- 10.1. Manage all data according to SOP B5.
- 10.2. Record anemometer installation dates and times, locations, positions, heights, orientations, analog output channel connections, wire color codes, initial test results, names of personnel conducting installation, anemometer model number and serial number, and other relevant information in the laboratory records.
- 10.3. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record with a bound record book designated for the method, which should contain copies of the electronic record. Overstrike all errors in writing with a single line; initial and date all such corrections.

11. Quality Control and Quality Assurance

- 11.1. Real-time data monitoring
 - 11.1.1. Configure the real-time data monitoring by defining rows from “11. Limit: Min” to “14. Email” (Table 2) for the analog signals that need to be monitored. Refer to SOP B2 for detailed descriptions of the configuration.
 - 11.1.2. With the configuration described in Section 11.1.1 and Table 2, signals acquired from the anemometer are converted to engineering units, and compared with pre-determined normal ranges (Table 1) every second by AirDAC. Alarms are shown immediately, and are sent via email by AirDAC to indicate any out-of-range data that might be collected (SOP B2).
- 11.2. Daily data inspection
 - 11.2.1. Plot collected anemometer data in daily figures using Air Data Pre-Processing (ADPP) software at any time after a day’s data is collected (SOP B3). Compare airflow data with the wind direction data obtained at the site using the roof-mounted vane anemometer (SOP E4). Verify that windward sides of the barn(s) show inward airflow in the sonic anemometer data during periods when the wind is blowing.
- 11.3. Physical inspection
 - 11.3.1. If the real-time monitoring or the daily data inspection indicates abnormal performance, conduct a visual inspection of the anemometer.
 - 11.3.2. Inspect the anemometer for any physical damage.
 - 11.3.3. Inspect the anemometer for any installation change, including its position, height, and orientation. Restore the anemometer to its original status if necessary.
 - 11.3.4. Inspect for obstructions to the acoustic path, including rain drops, ice, snow, or other debris. Clear the acoustic path if it is blocked.
- 11.4. Instrument check
 - 11.4.1. Perform the following instrument checks at the frequency specified in the QAPP.
 - 11.4.2. Standard inter-comparison: Compare the output of the anemometer with that from an unused, ‘standard’ anemometer.
 - 11.4.2.1. Locate the two anemometers next to each other on a calibration mount, verifying that their orientation is the same. This can be accomplished using a precision level and a compass to verify its vertical position and the orientation of the support arm, which should be directed to North.

- 11.4.2.2. Conduct measurements with both anemometers for 1 h.
- 11.4.2.3. Determine the paired differences between the measurements generated by the two anemometers, and compute the mean difference.
- 11.4.2.4. If the mean difference between anemometers is < 0.4 m/s (1% of full scale), then the anemometer being checked is in good working order.
- 11.4.2.5. If the mean difference between anemometers is > 0.4 m/s, then the anemometer being checked is defective. Return it to the manufacturer for repair or replacement.
- 11.4.3. In-field inter-comparison: Compare output of anemometers that are measuring air velocity in a common ventilation opening at times when all units should theoretically be reading the same air velocity (perpendicular wind direction, still air). This method is the one that will be applied most often.
 - 11.4.3.1. All anemometers are mounted in the same opening (e.g. ridge opening, sidewall opening) in identical fashion.
 - 11.4.3.2. Compare measurements taken over periods when wind is blowing directly into the sidewall and the air velocities should theoretically be the same.
 - 11.4.3.3. Compare the means and standard deviation of each anemometer. The means and standard deviations should be similar or have an explainable difference. If one of the anemometers produces outlying data as compared with the other anemometers in the opening, conduct a standard inter-comparison (Section 11.4.2) or switch with another anemometer and repeat the test. This latter option will rule out possible problems with the signal cables.
 - 11.4.3.4. If this in-field inter-comparison cannot be performed at the frequency specified in the QAPP, perform the standard inter-comparison method (Section 11.4.2) instead.

12. References

- 12.1. ASTM. 2001. Standard Test Method for Determining the Performance of a Sonic Anemometer/Thermometer. American Society for Testing and Materials, Philadelphia, PA, No. D6011-96.
- 12.2. Campbell Scientific (Canada) Corporation. 2001. RM Young 81000. <http://www.campbellsci.ca/CampbellScientific/Catalogue/81000.html>, accessed on 7/13/2006.
- 12.3. Foken, T., and B. Wichura. 1996. Tools for quality assessment of surface-based flux measurements. *Agric. Forest Meteorol.* 78, 83-105.
- 12.4. RM Young Company. 2005. Model 81000 Ultrasonic Anemometer, Rev C031405. Manual PN 81000-90. R. M. Young Co., Traverse City, MI.
- 12.5. SOP B1. 2006. Data Acquisition and Control Hardware. Standard Operating Procedure B1. Purdue Ag Air Quality Lab.
- 12.6. SOP B2. 2006. Air Quality Measurement Data Acquisition and Control software (AirDAC). Standard Operating Procedure B2. Purdue Ag Air Quality Lab.
- 12.7. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.

- 12.8. SOP E4. 2006. Wind Speed and Direction Measurements Using the R.M. Young Model 05103L Anemometer. Standard Operating Procedure E4. Purdue Ag Air Quality Lab.

13. Contact Information

- 13.1. Anemometer manufacturer:

R. M. Young Company

2801 Aero Park Drive

Traverse City, MI, 49686

Customer support and repair: 231-946-3980, Fax 231-946-4772

email: met.sales@youngusa.com

FAN STATUS MONITORING USING VIBRATION SENSORS

Standard Operating Procedure (SOP) A7

FAN STATUS MONITORING USING VIBRATION SENSORS

Standard Operating Procedure (SOP) A7

Prepared by

Matthew J. Darr (Ohio State Univ.) and Lingying Zhao (Ohio State Univ.)

Reviewed by

Ji-Qin Ni, Albert J. Heber and Bill W. Bogan

Effective Date: November 6, 2006

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions	3
4. Health and Safety	3
5. Cautions	3
6. Interferences	4
7. Personnel Qualifications.....	4
8. Equipment and Supplies	4
9. Procedures	4
10. Data and Records Management	5
11. Quality Control and Quality Assurance	5
12. References.....	5

1. Scope and Applicability

- 1.1. Accurate ventilation measurements are needed to obtain air emissions from barns.
- 1.2. Calculating fan airflow rate requires monitoring of fan status.
- 1.3. Sensing vibration of the fan is an effective fan monitoring strategy.
- 1.4. This method assumes that the ventilation fan vibrates sufficiently. It has been tested on 48" diameter belted fans but has not been tested on smaller fans and direct-drive fans.
- 1.5. This method has not been tested on variable speed fans.

2. Summary of Method

When a ventilation fan operates, propeller rotation causes the fan housing and hood structure to vibrate. This vibration disappears when the rotation stops. Therefore, the vibration and non-vibration of the fan housing correspond to the on and off status of the ventilation fan. A vibration sensor is used to detect this vibration and hence to monitor the on/off status of the fan. The vibration sensor is mounted directly to the fan housing or hood at the point where the vibration is sufficient to activate the sensor. The sensor provides logic zero and logic one outputs for the fan off and on states, respectively. A logic zero results if the belt breaks and the motor continues to operate because most of the vibration is derived from the rotating propeller. The logic signal is acquired by the data acquisition system in the instrument shelter every second, averaged every 15 s and 60 s, and saved in two separate data files, one for 15 s and another for 60 s data, with time stamps and other acquired measurement variables.

3. Definitions

- 3.1. Fan Hood: Fiberglass cover used to direct the exhaust air out of an agricultural fan. It is typically cantilevered off the side or end wall of a barn.
- 3.2. Vibration Sensor: Any sensor that can adequately detect the differences in vibration of a fan hood when the fan is and is not operating. Sensor should be of adequate quality to withstand the environmental conditions of external agricultural fans.

4. Health and Safety

- 4.1. Before mounting vibration sensor, make sure the fan power is disconnected.
- 4.2. Never stand directly in front of the fan when adjusting sensitivity of the vibration sensor.

5. Cautions

- 5.1. Rigidly mount the vibration sensor to the fan hood to ensure that mechanic vibration is transferred from the fan hood into the sensor (refer to Sections 9.1.2 - 9.1.4).
- 5.2. Carefully adjust the vibration sensor to allow the sensor to correctly pick up the fan operation signal. Fan size and type cause differences in vibration characteristics, thus the user must adjust individual sensors when they are installed in individual fan hoods (refer to Section 9.2.1).

- 5.3. Protect sensors from wet environments common to barns all year round (refer to Section 9.3.3).

6. Interferences

- 6.1. Other sources of mechanical vibration (other fans) near the fan may cause false positive readings. The sensor sensitivity should be adjusted to remove the false positives.
- 6.2. Malfunction of the data acquisition system will cause false signals to be recorded. Make sure that the data acquisition system is working properly.
- 6.3. Failure of the 5 VDC power supply to the vibration sensor will cause false negatives. Make sure that the sensors are properly powered.

7. Personnel Qualifications

- 7.1. Personnel should be trained by experienced researchers on the installation, adjustment, wire connection, and signal testing of the vibration sensor before initiating the procedure.
- 7.2. Each analyst must read and understand this SOP.

8. Equipment and Supplies

- 8.1. OSU-06 (Ohio State University, Columbus, Ohio) vibration sensors.
- 8.2. Data acquisition system to record status of each vibration sensor (refer to SOPs B1 and B2).
- 8.3. Electric drill and 9/64 inch drill bit to make holes in the fan hood.
- 8.4. Screws (#6 x 32) to fix the sensor to the fan hood. Choose a length suitable to clear the thickness of the fan housing but short enough that the screw will not interfere with the fan itself.
- 8.5. Lock washers, lock nuts, or thread lock to be used with the screws.
- 8.6. A screwdriver to drive in the screws.
- 8.7. A multimeter to check the sensor signal.
- 8.8. Sensor power supply and signal cable (3-conductor, 22 gage shielded).
- 8.9. Digital camera.

9. Procedures

- 9.1. Installation
 - 9.1.1. Determine the location of maximum vibration along the sidewall or bottom wall of the fan hood or fan housing by touching a hand to the hood and feel the vibration. Possible locations for sensor installation are shown in Fig. 1.
 - 9.1.2. Drill a 9/64 inch hole in the fan hood to accommodate a #6 x 32 mounting screw.
 - 9.1.2.1. Do not drill the mounting hole at the location where the fan propeller is inside the fan housing, as the rotating propeller might hit mounting screw head (Fig. 1).
 - 9.1.3. Securely fasten the vibration sensor to the sidewall using a #6 x 32 screw placed through the vibration sensor and the clearance hole in the sidewall.

- 9.1.4. Use either lock washers, lock nuts, or thread lock to ensure that the mounting screw will not loosen over long term exposure to vibration.
- 9.2. Calibration
 - 9.2.1. After installation, each vibration sensor output must be adjusted to match the sensor sensitivity with the mechanical vibration present at the fan housing or hood.
 - 9.2.1.1. Start the calibration with the fan running and the sensor outputting an off signal.
 - 9.2.1.2. Increase sensitivity until the sensor output signal indicates an on status.
 - 9.2.1.3. Stop fan and verify that the vibration sensor has returned to an off status state.
 - 9.2.1.4. Verify that other vibration sources such as adjacent fans, do not activate the sensor.
- 9.3. Connection to data acquisition module
 - 9.3.1. Connect the digital output signal from the vibration sensor to a digital input data acquisition board (refer to SOP B1).
 - 9.3.2. Supply 5 VDC to the vibration sensor from the data acquisition system. This power supply must be fused and provide a means of reverse polarity protection.
 - 9.3.3. Use weather tight connectors to prevent corrosion of electrical connections. If it is possible, install the sensor on the bottom side of the fan housing or hood to avoid direct rain and sun radiation.

10. Data and Records Management

- 10.1. Record the sensor make and model, locations of installation, data acquisition channel connections (refer to the measurement and control signal channel design in SOP B1), initial test results, and installation/testing personnel to the field notes spreadsheet of the project. Take digital photos as documentation if it is necessary.
- 10.2. Manage the data according to SOP B5 (Data Management).

11. Quality Control and Quality Assurance Section

- 11.1. Initial checks
 - 11.1.1. Upon installation, check all vibration sensors for appropriate operation and adjust their sensitivity as necessary.
- 11.2. Maintenance
 - 11.2.1. Compare the vibration sensor signal with the fan stage process relay signal and evaluate the discrepancies between the two signals.

12. References

- 12.1. SOP B1. 2006. Data Acquisition and Control Hardware. Standard Operating Procedure B1. Purdue Ag Air Quality Lab.
- 12.2. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure. Purdue Ag Air Quality Lab.
- 12.3. Ni, J.-Q, A.J. Heber, T.L. Lim, and C.A. Diehl. 2005. A low-cost technique to monitor ventilation fans at animal buildings. In Proceedings of the Seventh International Livestock Environment Symposium, 389 – 395. St. Joseph MI: ASAE.

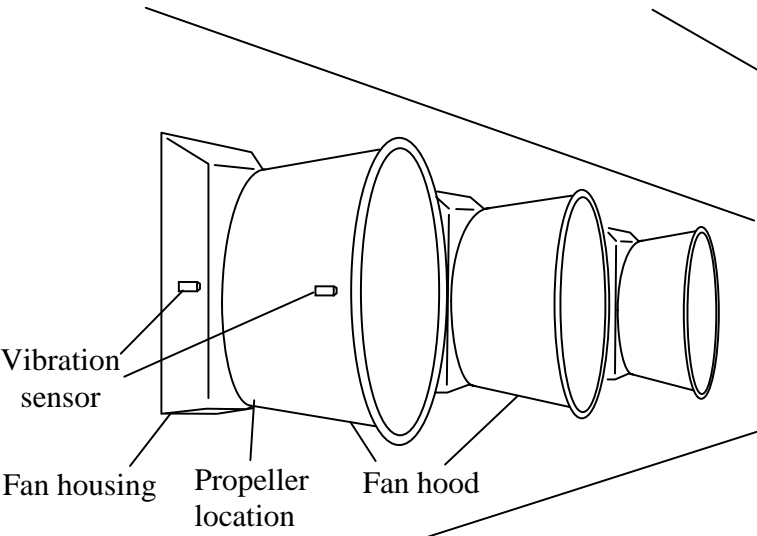


Figure 1. Locations of vibration sensors (Adapted from Ni et al., 2005). Each fan needs only one sensor.

**VENTILATION FAN AIRFLOW MEASUREMENTS USING
THE AIR VELOCITY TRAVERSE METHOD
Standard Operating Procedure (SOP) A8**

**VENTILATION FAN AIRFLOW MEASUREMENTS USING
THE AIR VELOCITY TRAVERSE METHOD
Standard Operating Procedure (SOP) A8**

**Prepared by
Teng-Teoh Lim**

**Reviewed by
Albert J. Heber and Bill W. Bogan**

Effective Date: November 6, 2006

**PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)
Agricultural and Biological Engineering, Purdue University
West Lafayette, IN 47907**

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions	3
4. Health and Safety	3
5. Cautions	4
6. Interferences	4
7. Personnel Qualifications.....	4
8. Equipment and Supplies	4
9. Procedures	5
10. Data and Records Management	7
11. Quality Control and Quality Assurance	7
12. References.....	7

1. Scope and Applicability

- 1.1. Accurate *in situ* measurements of ventilation fan airflow rates are needed to calculate emission rate.
- 1.2. A method is needed to conduct airflow tests on fans that cannot be analyzed using the Fans Assessment Numeration System (FANS, SOP A2) for the following reasons:
 - 1.2.1. Fans are located in areas that are physically inaccessible to the relatively large FANS analyzer.
 - 1.2.2. Fans are too large (> 54")
 - 1.2.3. Fans are too small (< 24")
 - 1.2.4. A proper seal can not be made between the FANS analyzer and the ventilation fan, due to obstructions
 - 1.2.5. FANS analyzer is not available
- 1.3. This method traverses the air velocity over the fan (airflow) cross-section at the inlet or outlet.
 - 1.3.1. The method is applicable to wall or pit ventilation fans of mechanically-ventilated barns.
 - 1.3.2. The method is applicable to single- or variable-speed fans.
 - 1.3.3. The method is applicable to all fan sizes (typically 10" to 60" diameter).
 - 1.3.4. This procedure takes approximately 10 min per fan to complete.

2. Summary of Method

The as-found (degraded) fan airflow capacity can be determined by multiplying the mean traverse velocity by the cross-sectional area of the measured airflow. To apply this method, it is necessary to divide the air stream cross-section into multiple sections of equal area. Air velocity is measured at the center of each section (Henderson and Perry, 1976). The accuracy of the calculated airflow rate increases as more sections are measured, because the airflow velocity profile is usually non-uniform. The fan operating pressure and fan speed should be held constant during the test. The velocity can be measured using a calibrated pitot tube and pressure transducer, a hot-wire anemometer, or a vane anemometer.

3. Definitions

- | | | |
|------|-------|--|
| 3.1. | dP | Differential pressure |
| 3.2. | FANS | Fans Assessment Numeration System |
| 3.3. | PAAQL | Purdue Agricultural Air Quality Laboratory |
| 3.4. | SOP | Standard Operating Procedure |

4. Health and Safety

- 4.1. Be careful when working close to high-speed fan blades.
- 4.2. Wear safety glasses while working around an operating fan.

5. Cautions

- 5.1. Avoid intervening with the barn environmental control system to adjust fan operating pressure to desired values, unless the barn is empty, or when proper ventilation airflow can be ensured for the comfort of the animals in the barn.
- 5.2. Air velocity meters have specific limitations:
 - 5.2.1. Hot-wire anemometers are non-directional.
 - 5.2.2. Vane anemometers cover larger measurement areas, thus limiting the number of traverse points for small ventilation fans.
 - 5.2.3. Pitot tubes require sensitive pressure transducers.

6. Interferences

- 6.1. High wind speeds around the barn can affect ventilation fan operation. Avoid conducting traverse measurements when wind speeds are >5 m/s (>11 mph).
- 6.2. Avoid removing fan shutter and guard when conducting the measurement.
- 6.3. Dust collected on the air velocity meter can reduce accuracy.
- 6.4. When the traverse measurement covers only a narrow range of differential pressure (dP), comparison with the full fan performance curve is not possible. This can result in greater uncertainty of the calculated airflows.
 - 6.4.1. If possible, measure airflow at several dPs (by turning other fans on or off to increase or decrease the overall dP), to span the more of the operating range. However, avoid intervening with the environmental control system unless the conditions in Section 5.1 can be met.
- 6.5. Avoid standing in or blocking the air stream during the test, as this will affect the flow profile.
- 6.6. Airflow at the exit side of the ventilation fan is more turbulent than at the fan inlet, which can reduce the accuracy of velocity measurements. If possible, conduct the air velocity traverse at the fan inlet.

7. Personnel Qualifications

- 7.1. Personnel should understand barn ventilation systems and static pressure controls, and be able to identify the normal ranges of barn dP and fan performance.
- 7.2. Each analyst must read and understand this SOP and the applicable instrument manual(s), and be trained in the use of the velocity meter before initiating the procedure.

8. Equipment and Supplies

- 8.1. Air velocity meter
 - 8.1.1. Portable vane anemometer (Model 451126, Extech, Bohemia, NY, or equivalent)
 - 8.1.2. Hot-wire anemometer (Model 444, Kurz, Inc., Monterey, CA, or equivalent)
 - 8.1.3. Pitot tube and pressure sensor (Model 267 Differential Pressure Transducer, Setra Systems, Boxborough, MA)
- 8.2. Stopwatch

9. Procedures

- 9.1. Determine the number and locations of traverse points.
 - 9.1.1. When choosing traverse locations, consider factors including safety, accessibility, flow turbulence (swirling airflow), and airflow blockage.
 - 9.1.2. For a square or rectangular duct or inlet area, divide the cross-section into equal squares or rectangles.
 - 9.1.2.1. Use at least 16 traverse points.
 - 9.1.3. For round ducts or fan shrouds, measure at least two axes (horizontal and vertical) of traverse points. The minimum number of traverse points is eight (8) points per axis. Fig. 1 shows an example in which 10 points per axis may be measured.

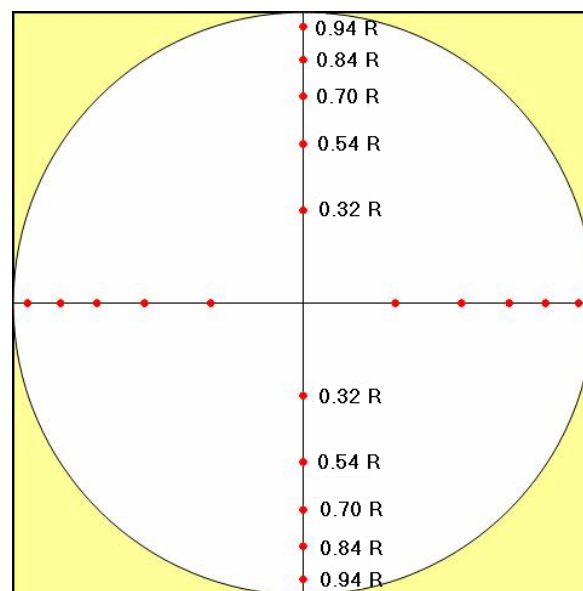


Figure 1. Example of 10 traverse points per axis, with traverse point distances as a percentage of diameter. The entire traverse includes both vertical and horizontal axes, for a total of 20 points. Distances are given as fractions of radius (R), starting at the center.

- 9.1.3.1. Apply the following equation to determine the location of each traverse point on a radius, from the center of the duct:

$$L_i = \sqrt{\frac{(i-0.5)R^2}{n}} \quad (1)$$

Where:

- L_i Distance to the i^{th} traverse point on a radius, from the center
- i i^{th} traverse point on a radius, from the center
- R Radius of the total cross-sectional area of airflow
- n Total number of traverse points on a radius

9.2. Measure air velocity at each point.

- 9.2.1. Attach the pitot tube, hot-wire tip or rotating vane to a probe (if the device does not have a long enough probe for the fan) that is sufficiently long to allow the operator to stand outside the airflow area, to avoid airflow blockage.
- 9.2.2. Mark the probe with unit measurements, or with the distances of the desired traverse points with respect to a fixed fan fixture or wall, so that the exact locations can be readily established during the traverse without obstructing the flow.
- 9.2.3. Maintain the fan operating pressure and speed.
 - 9.2.3.1. Manipulate fan controls as necessary to “freeze” the fan speed.
 - 9.2.3.2. Manipulate inlet controls as necessary to “freeze” the inlet openings.
- 9.2.4. Use a stopwatch to ensure the same measurement time for each traverse point, and use an air velocity meter or pressure sensor (for pitot tube measurement) equipped with an averaging function. Report all measurement results as velocity (m/s).
- 9.2.5. Record the date, time, analyst, barn, fan and other information. An example of a field record sheet is given in Table 1:

Table 1. Example of fan traverse field data sheet.

Date		Analyst	
Measurement site		Facility name	
Fan number		Fan size	
Traverse location		Traverse points	
Air meter S/N		Calibration date	
Notes			
Results			
Traverse location #*	Distance*	Velocity, m/s	Note

* Include a drawing that clearly defines the traverse locations and distances.

9.3. Calculate the airflow rate

- 9.3.1. Calculate the average velocity (m/s) of the traverse points.
- 9.3.2. Calculate the cross-sectional flow area (m²).
- 9.3.3. Calculate the fan airflow rate (m³/s) by multiplying the mean velocity by flow area.

10. Data and Records Management

- 10.1. Maintain laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
- 10.2. Manage all data according to SOP B5.
- 10.3. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes. Overstrike errors in writing with a single line. Initial and date all corrections.

11. Quality Control and Quality Assurance

- 11.1. Verify the traverse values in a laboratory equipped with a fan test facility or compare the mean velocity to calibrated, portable fan tester data (SOP A2).
- 11.2. At the beginning and end of each set of field measurements, perform a “zero air speed” measurement to make sure the anemometer reads zero. If the anemometer appears to be malfunctioning, have it checked by the manufacturer.
- 11.3. Have the air velocity measurement devices calibrated and certified by manufacturers annually.
- 11.4. Worn or improperly-functioning backdraft shutters can alter the velocity profile and affect the fan capacity. Inspect and verify the integrity of the shutter, motor, and blades, and note them in the field notes.

12. References

- 12.1. Henderson, S. M., and R. L. Perry. 1976. Velocity measurement. In *Agricultural Process Engineering*, 57-60. Westport, Ct.: AVI Publishing.
- 12.2. SOP A1. 2006. Laboratory Testing of Ventilation Fan Performance. Standard Operating Procedure A1. Purdue Ag Air Quality Lab.
- 12.3. SOP A2. 2006. Measurement of Ventilation Fan Flowrate Using the Fan Assessment Numeration System (FANS). Standard Operating Procedure A2. Purdue Ag Air Quality Lab.
- 12.4. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.

**SOP A9. STANDARD OPERATING PROCEDURE
FOR MEASUREMENT OF AIRFLOW RATE OR CALIBRATION OF AIR
SAMPLING
INSTRUMENTS WITH GILIBRATOR 2 CALIBRATION SYSTEMS**

**SOP A9. STANDARD OPERATING PROCEDURE
FOR MEASUREMENT OF AIRFLOW RATE OR CALIBRATION OF AIR SAMPLING
INSTRUMENTS WITH GILIBRATOR 2 CALIBRATION SYSTEMS**

Prepared by

Hua Xu

Reviewed by

Teng-Teoh Lim and Albert J. Heber

Effective Date: November 5, 2006

**PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)
Agricultural and Biological Engineering, Purdue University
West Lafayette, IN 47907**

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions of Terms and Acronyms	3
4. Health and Safety	4
5. Cautions.....	4
6. Personnel Qualifications	4
7. Equipment and Supplies	4
8. Procedures.....	5
9. Data and Records Management.....	6
10. Quality Control and Quality Assurance.....	6
11. References	6
12. Contact Information.....	7

1. Scope and Applicability

- 1.1. Contaminant emission studies require the use of various flow controllers and flow meters. Frequent verification and calibration are needed to ensure functionality and data quality. The flow controllers and measurement devices may include:
 - 1.1.1. Mass flow meter in the PAAQL Gas Sampling System (SOP G1).
 - 1.1.2. Mass flow controllers of PM sampling and monitoring devices.
 - 1.1.3. Mass flow meters in the sampling trains for collection of sorbent tube (SOP V1), canister (SOP V2), and impinger (SOP V3) samples for VOC analyses.
- 1.2. The Gillian Gilibrator 2 Calibration System is useful for measurement or calibration of air sampling equipment as a primary standard. This SOP is written only for the Gilibrator 2 Calibration System.
 - 1.2.1. The Gilibrator 2 is a high accuracy, electronic flow meter that provides instantaneous airflow readings and cumulative averaging of multiple samples.
 - 1.2.2. The three interchangeable Wet Cell Assemblies provide an overall range of flow rates from 1 cc/m to 30 L/min using the following flow cells:
 - 1.2.2.1. Low flow cell (1 to 250 cc/min)
 - 1.2.2.2. Standard flow cell (20 cc/min to 6 L/min)
 - 1.2.2.3. High flow cell (2 to 30 L/min)

2. Summary of Method

The purpose of the method is to measure airflow rate or calibrate air sampling equipment using the Gillian Gilibrator 2 Calibration System as a primary standard. A primary standard airflow measurement is a volume (V , measured volume of space between two infrared sensors) divided by a time interval (t , the time interval needed for the soap bubble film to travel between the two sensors that bound the volume) as performed by the control unit of the Gilibrator 2. Therefore, the flow rate is the volume per unit of time (V/t), which is calculated and then displayed directly on the unit's Liquid Crystal Display (LCD).

3. Definitions of Terms and Acronyms

AC	Alternating current
cc	Cubic centimeters
LCD	Liquid crystal diode
LPM	Liters per minute
NIST	National Institute of Standards and Technology
PAAQL	Purdue Agricultural Air Quality Lab
PM	Particulate matter
RMA	Returned material authorization
SOP	Standard operating procedure

4. Health and Safety

- 4.1. Be careful when working with electrical power connections.
- 4.2. Do not pressurize the cell assembly. Excessive pressure may cause the cell to rupture, resulting in personal injury.

5. Cautions

- 5.1. Do not remove, cover or alter any labels or tags on the instrument.
- 5.2. Batteries must be fully charged (usually for 14 h) for the instrument to function properly.
- 5.3. Always turn off the instrument before changing any batteries.
- 5.4. Use only the manufacturer's soap solution (Part No. 800450) with the wet flow cell assembly. Use of other soap solution may damage the flow cell.
- 5.5. Do not overfill the soap solution reservoir.
- 5.6. Do not attempt to repair or modify the instrument. If repair is needed, contact the Sensidyne Service Department to arrange for a Returned Material Authorization (RMA).
- 5.7. When the wet cell assembly will not be used for a long period of time, install rubber storage tubing between the inlet and outlet bosses to prevent the soap solution from evaporating, as this would change the soap concentration.
- 5.8. When a wet cell assembly is to be transported by air, disconnect the storage tubing from either the air outlet boss (upper) or air inlet boss (lower). Otherwise, the flow cell assembly could become over-pressurized, possibly causing a rupture within the bubble generator.
- 5.9. Do not transport unit with soap solution or storage tubing in place.
- 5.10. Always mount or remove the cell assembly by grasping and rotating the lower part of the assembly.
- 5.11. Never lean on the bubble generator to press the plate into place. Placing excessive weight on the cell assembly may break the instrument.
- 5.12. Never use alcohol, acetone or any other harsh solvent or cleaner to clean the bubble generator cell.

6. Personnel Qualifications

- 6.1. Personnel should be trained in the use of the instrument before initiating the procedure.
- 6.2. Each analyst must read and fully understand the entire instrument manual and this SOP before operating or maintaining the instrument.

7. Equipment and Supplies

- 7.1. Flow calibration system (Gilian Gilibrator 2, Sensidyne Inc, Clearwater, FL), consisting of the following components (Fig. 1):

- 7.1.1. Control unit base
- 7.1.2. Wet cell assemblies:
 - 7.1.2.1. Low flow cell (1 to 250 cc/min)
 - 7.1.2.2. Standard flow cell (20 cc/min to 6 L/min)
 - 7.1.2.3. High flow cell (2 to 30 L/min)
- 7.1.3. AC charger
- 7.1.4. Tubing (including rubber storage tubing)
- 7.1.5. Soap solution and dispenser

8. Procedures

8.1. Assembly

- 8.1.1. Connect the battery charger to the charger jack in the control unit and to an appropriate AC outlet. Make sure the batteries are fully charged (≥ 14 h) before operating.
- 8.1.2. Mount the wet cell assembly according to Figs. 2 and 3.
 - 8.1.2.1. Position the control unit base on a flat level surface.
 - 8.1.2.2. Select the appropriate wet cell assembly (low, standard, or high flow) according to measurement requirements.
 - 8.1.2.3. Grasp the cell assembly on the lower portion, and move it to the mounting plate on the control unit.
 - 8.1.2.4. Align the mounting pins of the flow cell with the mounting plate slot. Make sure the bottom of the flow cell mounts flush with the mounting plate.
 - 8.1.2.5. Rotate the cell assembly clockwise until it clicks into place. The front cell assembly should face toward the display on the control unit.
- 8.1.3. Add soap solution to the wet cell assembly.
 - 8.1.3.1. Remove the storage tubing from the air outlet boss (upper).
 - 8.1.3.2. Using the storage tubing as a funnel, slowly add the soap solution to the reservoir from the dispenser.
 - 8.1.3.3. Fill until the solution level reaches the angled edge at the bottom of the bubble generator ring.
- 8.1.4. Connect the instrument (a flowmeter or an air sampling pump) that is to be checked or calibrated to the Gilian Gilibrator 2 Calibration System.
 - 8.1.4.1. If the flow of the instrument to be checked or calibrated is positive, connect the tubing to the lower boss.
 - 8.1.4.2. If the flow of the instrument to be checked or calibrated is negative, connect the tubing to the upper boss.

8.2. Operation

- 8.2.1. Start-up
 - 8.2.1.1. Turn on the sampling source.
 - 8.2.1.2. Push the "Bubble Initiate" button several times to wet the inner walls of the flow tube.
 - 8.2.1.3. Press the "ON" button on the control unit, and wait about 10 s for the unit to self-test.

8.2.2. Bubble generation

- 8.2.2.1. Push the “Bubble Initiate” button again, and hold until the first bubble is initiated and travels up to the flow tube.
- 8.2.2.2. A timing sequence is initiated when the rising bubble passes the lower sensor.
- 8.2.2.3. The timing sequence is completed when the bubble passes the upper sensor.
- 8.2.2.4. The timing information is transmitted to the control unit, where flowrate is calculated and then displaced on the LCD (Fig. 4).
- 8.2.2.5. Release the button and initiate a second bubble to travel up to the flow tube.
- 8.2.2.6. Repeat this procedure to obtain additional readings.

8.2.3. Flow readout

- 8.2.3.1. The measured flow rate of the last test, the cumulative average flow rate across tests, and the current test/sample number are displayed on the LCD of control unit (Fig. 4).
- 8.2.3.2. Press the DELETE/RESET button for ~ 1 s to delete an obviously false reading.
- 8.2.3.3. Press the DELETE/RESET button for ~ 3 s to reinitiate the entire sequence.

8.3. Calibration

- 8.3.1. The instrument should only be calibrated by the manufacturer.

9. Data and Records Management

- 9.1. Always observe the individual measurements for consistent readings.
- 9.2. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
- 9.3. Manage all data according to SOP B5.
- 9.4. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes. Overstrike all errors in writing with a single line, and initial and date all such corrections.
- 9.5. Keep a record of the calibration certificate, and make sure the calibration record (expiration date) is shown on the calibration system.

10. Quality Control and Quality Assurance

- 10.1. Conduct general and additional wet cell maintenance as per the instrument manual.
- 10.2. Return the instrument to the manufacturer annually for calibration.
- 10.3. All devices should be certified for accuracy, and traceable to the National Institute of Standards and Technology (NIST).

11. References

- 11.1. Gilibrator Manual. 1998. Gilian Gilibrator 2 Calibration System Operation & Service Manual. Revision G (Document No. 850190M). Sensidyne, Inc., Clearwater, FL.
- 11.2. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 11.3. SOP G1. 2006. The PAAQL Gas Sampling System. Standard Operating Procedure G1.

- Purdue Ag Air Quality Lab.
- 11.4. SOP V1. 2006. Sampling of Volatile Organic Compounds (VOCs) in Air Samples through Use of Sorbent Tubes. Standard Operating Procedure V1. Purdue Ag Air Quality Lab.
 - 11.5. SOP V2. 2006. VOC Sampling Using Canisters. Standard Operating Procedure V2. Purdue Ag Air Quality Lab.
 - 11.6. SOP V3. 2006. Collection of Organic Amines in Air Samples Through Use of Sulfuric Acid-Containing Impingers. Standard Operating Procedure V3. Purdue Ag Air Quality Lab.

12. Contact Information

- 12.1. Manufacturer:
Sensidyne, Inc.
16333 Bay Vista Drive
Clearwater, Florida 33760 USA
<http://www.sensidyne.com/>
- 12.2. PAAQL: odor@purdue.edu, heber@purdue.edu, <http://www.AgAirQuality.com>

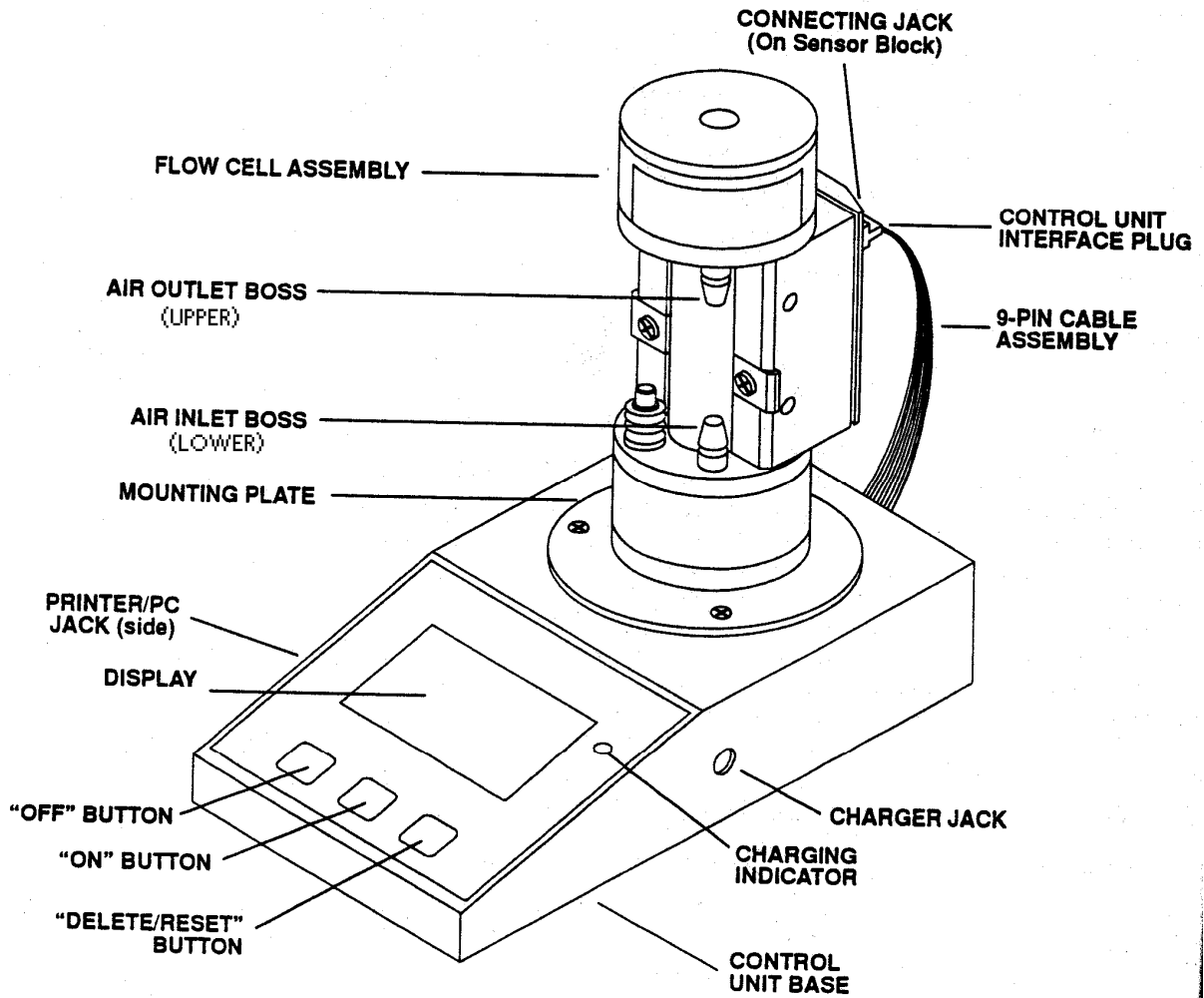


Figure 1: The Gilian Gilibrator 2 Calibration System.

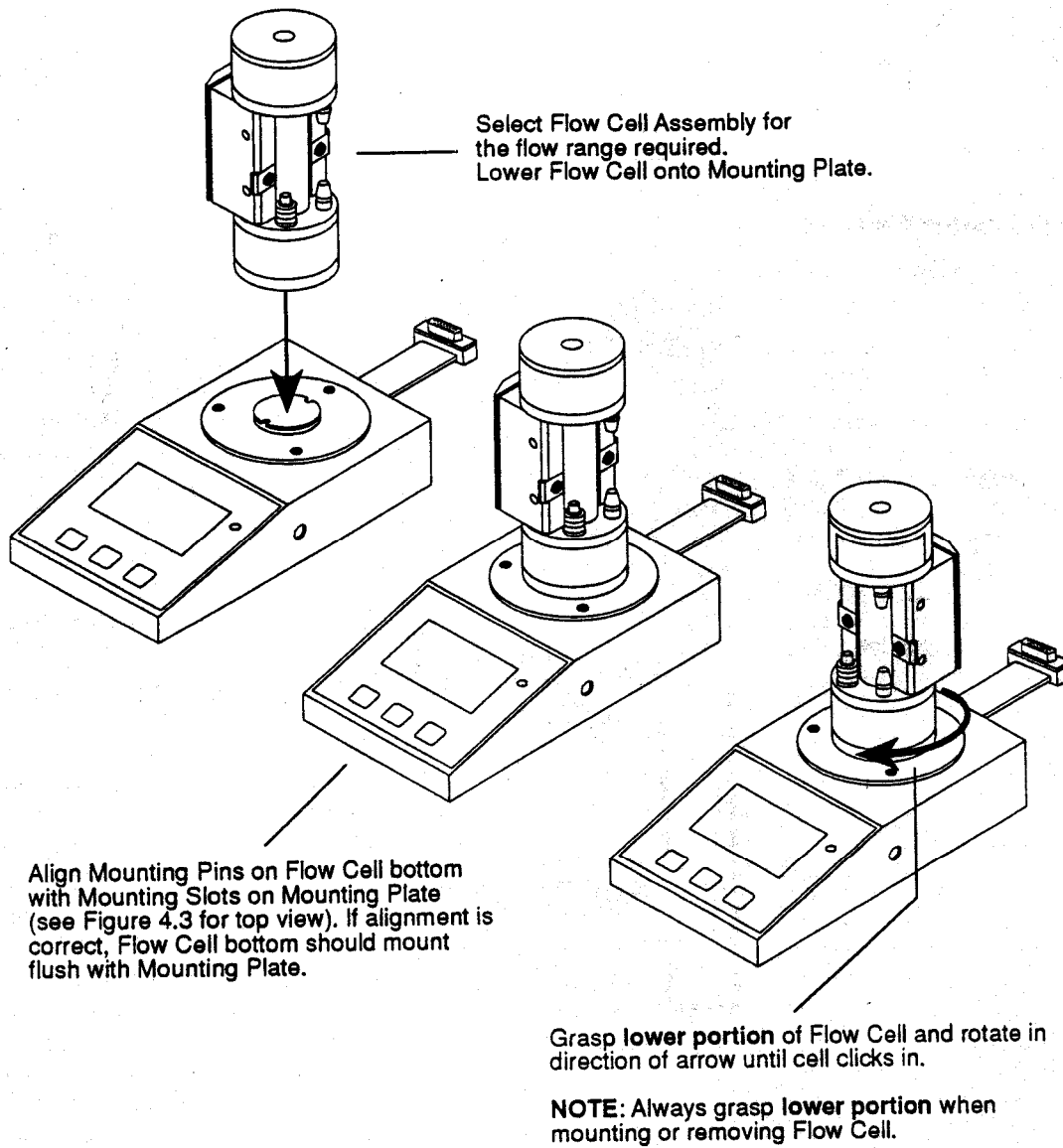


Figure 2: Flow Cell Mounting (3D View).

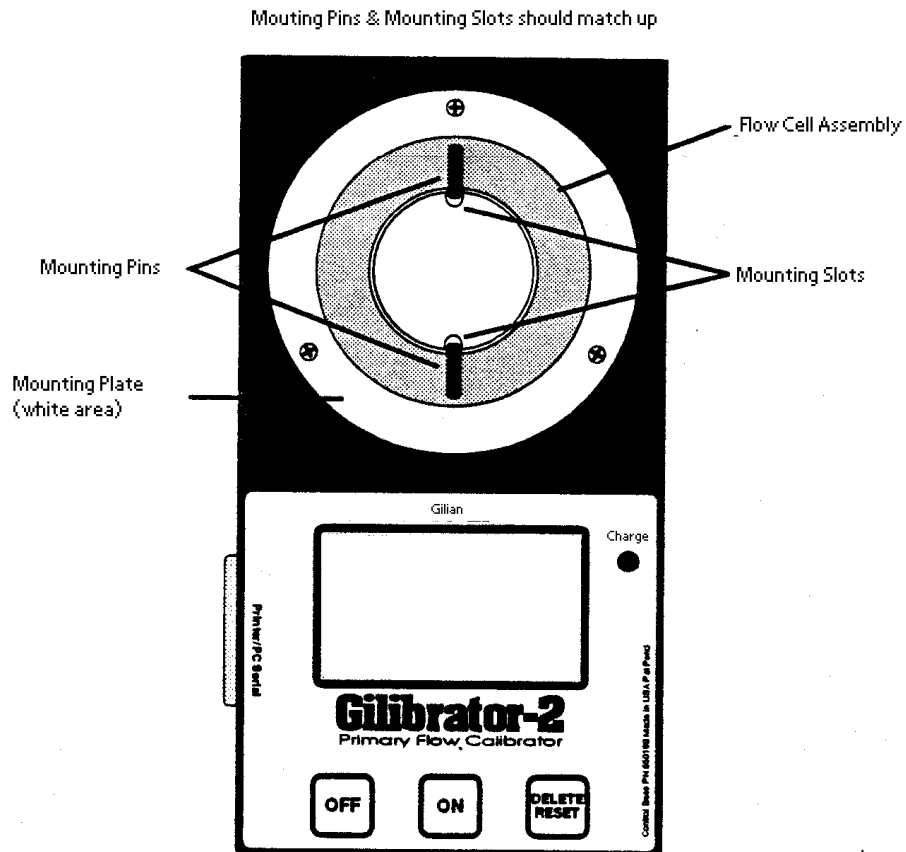


Figure 3: Flow Cell Mounting (Top View).

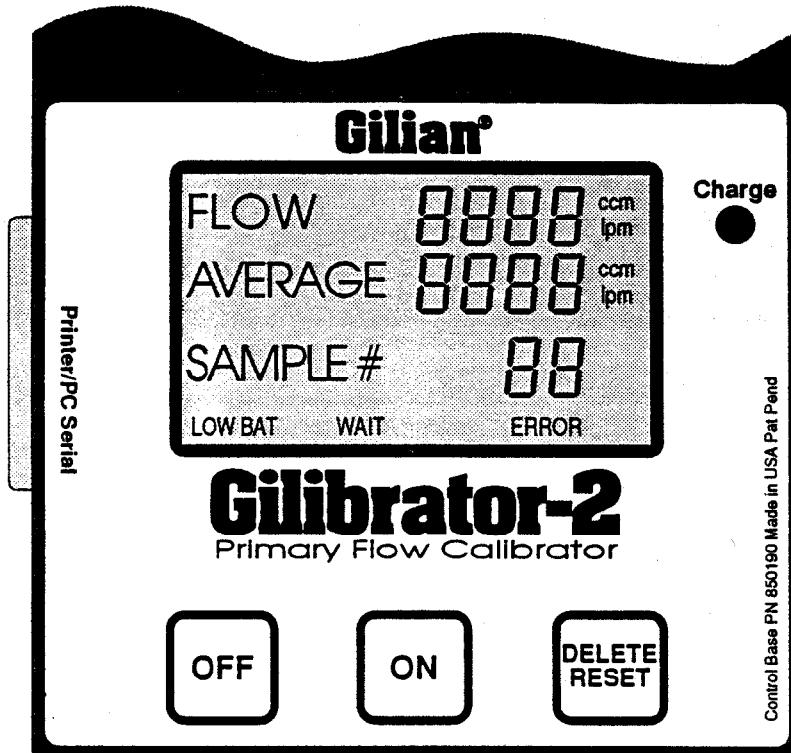


Figure 4: Gilian Gilibrator 2 Liquid Crystal Display.

FAN STATUS MONITORING USING RPM SENSORS

Standard Operating Procedure (SOP) A11

FAN STATUS MONITORING USING RPM SENSORS

Standard Operating Procedure (SOP) A11

Prepared by

Erin L. Cortus and Bill W. Bogan

Reviewed by

Albert J. Heber

Effective Date: December 28, 2007

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions	3
4. Health and Safety	4
5. Cautions	4
6. Interferences	4
7. Personnel Qualifications.....	5
8. Equipment and Supplies	5
9. Procedures	6
10. Data and Records Management	9
11. Quality Control and Quality Assurance	10
12. References	10

1. Scope and Applicability

- 1.1. Accurate fan airflow measurements are needed to obtain air emissions from mechanically ventilated livestock barns.
- 1.2. Calculating fan airflow rate requires monitoring of fan status.
- 1.3. Monitoring the rotational speed of a fan increases the accuracy of the flow calculation because fan airflow is directly proportional to fan speed at a given fan static pressure.
- 1.4. RPM sensors provide measurements of both operational status and rotational speed; thus, they are an effective fan monitoring strategy.
- 1.5. This method is applicable to any size of ventilation fan, and to both single-speed and variable-speed fans.
- 1.6. This method is implemented using solid-state Hall-effect sensors, which produce a signal when a magnet is in close proximity to the sensor. When the magnet is attached on a rotating part of the fan to monitor speed, the application is to measure rotational speed of the fan.
- 1.7. The rotational speed of the fan is expressed as revolutions per minute (RPM). Therefore, the Hall-effect sensor will be referenced as an RPM sensor in this method.
- 1.8. This method will focus on the use and installation of threaded barrel proximity sensors (Model MP100701, Cherry Corporation, Pleasant Prairie, WI) and threaded barrel gear tooth sensors (Model GS100701, Cherry Corporation, Pleasant Prairie, WI).

2. Summary of Method

An RPM sensor detects the rotational speed of an individual ventilation fan impeller or an associated part such as a pulley or pulley shaft. This method uses Hall-effect proximity sensors, which are solid-state devices that operate by monitoring the electronic signal created by a changing magnetic field. In simplified terms, when the sensor detects a magnetic field, a voltage is electromagnetically induced in the sensor's circuitry that causes a transistor to activate and produce an output signal. The output signal changes as the magnetic field changes due to the rotating magnet or metal part passing near the fixed sensor. A pulse in the output signal is created each time the magnet passes the sensor. The speed of the rotation is determined by the number of pulses in a time interval. The MP100701 proximity sensor (Cherry Corporation, Pleasant Prairie, WI) is a Hall-effect sensor that senses an external magnet that is attached to the rotating part. A gear tooth sensor, such as the GS100701 sensor (Cherry Corporation, Pleasant Prairie, WI) incorporates a stationary magnet within the sensor which detects the proximity of a moving discontinuous ferrous material. Hall-effect sensors have no moving parts within the sensor and are not susceptible to dust and environmental contamination.

3. Definitions

- | | |
|------------------|--|
| 3.1. DAQ | Data acquisition |
| 3.2. Fan Hood: | Fiberglass cover used to direct the exhaust air out of an agricultural fan. It is typically cantilevered off the side or end wall of a barn. |
| 3.3. Hall effect | Voltage induced across a current-carrying material when it is |

- 3.4. MCC perpendicular to a magnetic field
Measurement Computing Corporation
- 3.5. Tachometer Device that measures rotational speed
- 3.6. RPM Revolutions per minute
- 3.7. USB Universal serial bus

4. Health and Safety

- 4.1. Make sure the fan power supply is disconnected before installing RPM sensors,. If the power disconnect is not located near the fan, post signage at the power disconnect location to warn others not to turn the fan on.
- 4.2. Be careful when working near operational ventilation fans. The high-speed fan blades = hazardous, especially if they are not protected by fan shutters.
- 4.3. Be wary that wind can cause the fan blades to suddenly start spinning. Fix the fan blades in place before working in and around the fan.
- 4.4. Wear safety glasses while working near operating fans. Do not wear loose-fitting or baggy clothing, or dangling jewelry around an operational fan, and tie up long hair.
- 4.5. Protect your eyes and face from dust when working around the fan. Sudden dust clouds can form when the fans are turned on.
- 4.6. Ensure that magnets are not left in areas where animals may access them. If swallowed, magnets can cause serious digestive system problems.

5. Cautions

- 5.1. Excess moisture in the environment can enter the sensor housing where the wire leads are attached. In environments where the sensors may be frequently splashed with water (i.e. farrowing barns), apply room temperature-vulcanizing (RTV) silicone or its equivalent around the wire leads to increase resistance to water damage.
- 5.2. The sensors are operated in close proximity to a moving target. Ensure that the sensor is securely fastened to the sensor support and all wires are securely fastened away from moving parts to avoid damage to both the sensor and fan.
- 5.3. When using a magnet target, ensure that the magnet is securely set in place before turning on the fan.
- 5.4. The RPM sensor is susceptible to electrical surges caused by lightning. Proper grounding of the barn is recommended to minimize the risk of lightning damage.

6. Interferences

- 6.1. Malfunctioning of the data acquisition (DAQ) system will cause false signals to be recorded. Make sure that the DAQ system is working properly.
- 6.2. Make sure the signal wires from the RPM sensor are firmly attached and tidy to avoid being blown loose over time.
- 6.3. Signal noise can interfere with the RPM measurements. Using shielded multi-conductor cable and grounding the shield wire to the ground on the DAQ board can often alleviate the noise in the signal.

- 6.4. The sensing target should remain consistent in strength and location during the operation of the fan. Avoid placing the target near the edge of a fan blade, where the position of the target relative to the sensor may change with speed.
- 6.5. Poor signals may result if the gap between the sensor and sensor target is too large. Preliminary testing can determine the allowable gap distance.
- 6.6. When monitoring the rotation of a pulley wheel for belt-driven fans, monitor the rotation of the fan pulley, and not the drive pulley. Besides turning at different speeds due to different diameters, any belt slippage between the pulleys leads to overestimated fan rotational speeds when the drive pulley is monitored.
- 6.7. AirDAC determines the rotational speed on a per-second basis (i.e. X number of turns per second) and reports this value in rotations per minute; thus, the 1-s readings in RPM will increment by 60 RPM for every revolution increase per second. The one-minute average RPM values are a more accurate measurement of RPM.

7. Personnel Qualifications

- 7.1. Personnel should be trained by experienced researchers on the installation, adjustment, wire connections, and signal testing of the RPM sensor before initiating the procedure.
- 7.2. Each analyst must read and understand this SOP.

8. Equipment and Supplies

- 8.1. RPM sensors. Sensors covered under this SOP include the following:
 - 8.1.1. Threaded-barrel magnetic proximity sensors (Cherry part #MP100701 or equivalent)
 - 8.1.2. Threaded-barrel gear tooth sensors (Cherry part #GS100701 or equivalent)
- 8.2. Sensor target.
 - 8.2.1. Magnet (for Model MP100701 or equivalent only). Magnets can be purchased at craft and hobby supply stores and electronics stores.
 - 8.2.2. Surface of discontinuous ferrous material (for Model GS100701 or equivalent only). This may be the spokes of a fan pulley, or a bolt attached to a rotating surface.
 - 8.2.3. Quick-set epoxy.
- 8.3. Sensor mounting supplies. The specific supplies will vary based on the installation method, but may include brackets, cable ties, bolts, etc.
- 8.4. DAQ system (SOPs B1 and B2) to record signals from each vibration sensor
 - 8.4.1. USB-4303 Counter/Timer (Measurement Computing Corporation, Middleboro, MA)
- 8.5. Multimeter
- 8.6. Signal cable (3-conductor, 22 gage shielded)
- 8.7. Portable tachometer
- 8.8. Digital camera (optional)
- 8.9. Pull-up resistor (optional)

9. Procedures

9.1. Initial testing

- 9.1.1. Test the RPM sensor and its reaction to the proposed sensor target (i.e. magnet) to determine the mounting location and method.
- 9.1.2. Connect the sensor to a 5-VDC power supply and the output wire to the multimeter.
- 9.1.3. Vary the distance between the sensor and the target, and monitor the signal output using the multimeter. Determine the maximum gap that ensures a signal is induced.

9.2. Sensor installation

- 9.2.1. Turn off the fan and fix the fan blade in place to prevent the wind from turning the blade. Put up signs to inform others that you are working with the fan, if necessary.
- 9.2.2. Inspect the fan(s) to be monitored, and evaluate different mounting locations, based on the fan configuration and the maximum allowable distance between sensor and target (Section 9.1). A sensor bracket may be required to hold the sensor in position. Potential installation methods are described below, but others may be available.
 - 9.2.2.1. Mount the sensor to face a moving target on the fan shaft (Fig. 1).



Figure 1. RPM sensor mounted perpendicular to fan shaft.

- 9.2.2.2. Mount the sensor to face the fan pulley. The target may be either the pulley spoke for a gear tooth sensor, or a target attached to the pulley (Fig. 2).
- 9.2.2.3. Mount the sensor to face a target attached to a fan blade. The target should be as close to the center of the fan as possible. The sensor may be mounted facing the back or the front of the fan blade.

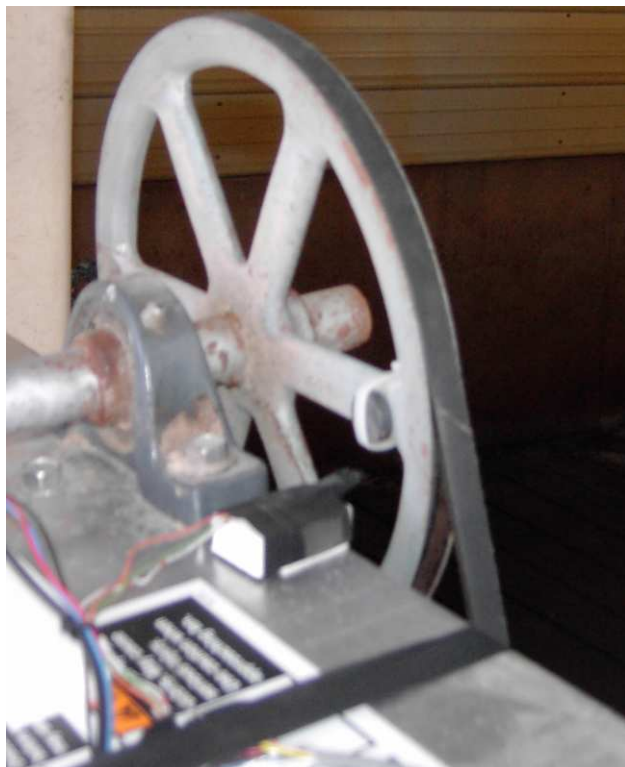


Figure 2. RPM sensor mounted perpendicular to fan pulley.



Figure 3. RPM sensor mounted to fan grating, perpendicular to fan blade.

9.3. Sensor target installation (for magnet targets only)

- 9.3.1. Clean the surface where the target will be attached (Figs. 1-3). If necessary, use sandpaper to roughen the surface.
- 9.3.2. Apply epoxy to the target and surface according to product directions.
- 9.3.3. Allow epoxy to set before starting the fan.

9.4. Connection to DAQ module

- 9.4.1. The wire leads for the sensor are described in Fig. 4. The pull-up resistor is not required when using the MCC USB-4303.

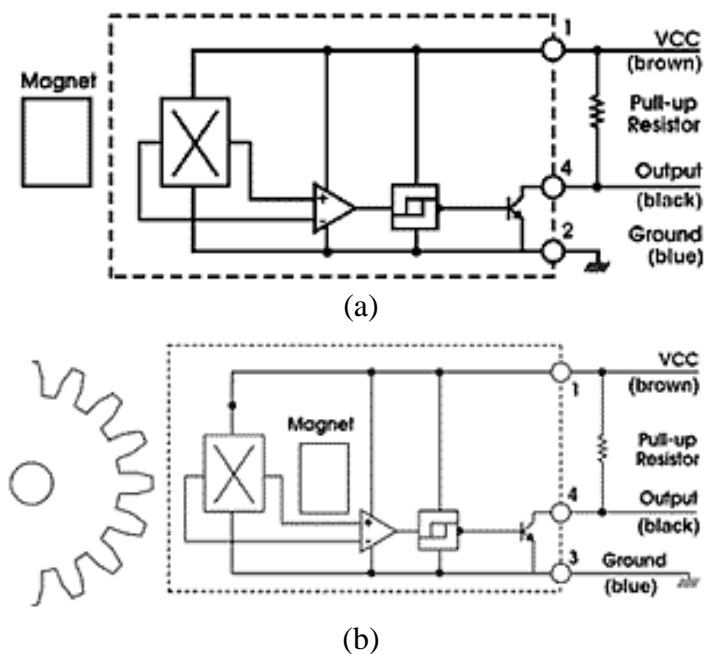


Figure 4. Block diagram and wire leads for Models MP1007 (a) and GS1007 (b) sensors (Cherry, 2007).

- 9.4.2. Connect the VCC (Brown) wire lead to a 5-VDC power supply, either on the USB-4303 counter module (Fig. 5; +5VDC), or on the DAQ board.
- 9.4.3. Connect the Output (Black) wire lead to a channel on the USB-4303 counter module (Fig. 5; 1INP1 to 1INP5 or 2INP1 to 2INP5).
- 9.4.4. Connect the Ground (Blue) wire lead to the ground of the USB-4303 counter module (Fig. 5, GND), or a ground connection on the DAQ board.
- 9.4.5. AirDAC settings. Enter a scalar value for the slope if the sensor detects multiple targets per revolution. For example, enter a slope of 0.25 if a gear-tooth sensor detects four fan pulley spokes each revolution.

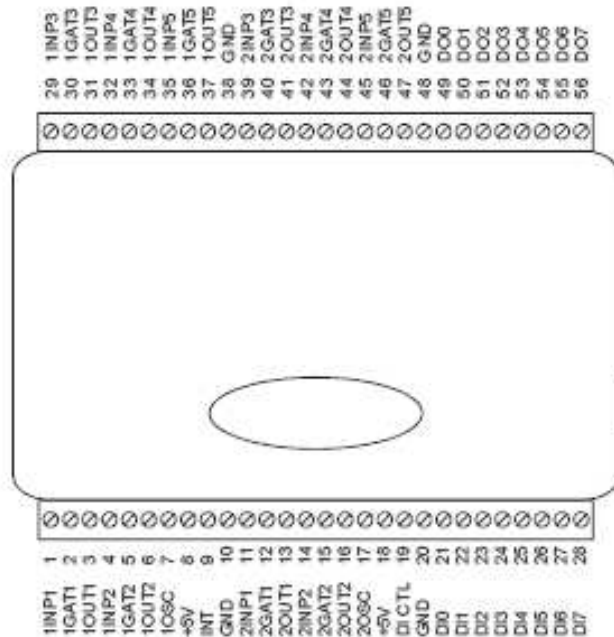


Figure 5. USB-4303 terminal descriptions (MCC, 2007)

9.5. Calibration checking with tachometer

- 9.5.1. Following the tachometer instructions, measure the rotational speed of the fan blade(s) using the appropriate targets for the tachometer. If necessary, adjust the tachometer measurement for the number of sensing targets per revolution.
- 9.5.2. Compare the tachometer measurements to the RPM sensor measurement.

9.6. Computer hardware & software

- 9.6.1. AirDAC (SOP B2)
- 9.6.2. ADPP (SOP B3)
- 9.6.3. CAPECAB (SOP B6)

10. Data and Records Management

- 10.1. Maintain laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
- 10.2. Record the sensor make and model, locations of installation, DAQ channel connections (refer to the measurement and control signal channel design in SOP B1), initial calibration check results, and installation/testing personnel to the field notes spreadsheet of the project. Take digital photos as documentation if necessary.
- 10.3. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes.
- 10.4. Manage the data according to SOP B5.

11. Quality Control and Quality Assurance

11.1. Initial checks

11.1.1. Upon installation, check all RPM sensors against the tachometer for appropriate operation, and adjust their positioning as necessary.

11.2. Periodic checks

11.2.1. Check all RPM sensors against the tachometer for appropriate operation at the QAPP-specified frequency, and adjust their positioning as necessary.

12. References

- 12.1. Sensors – Cherry Sensors FAQ. Online at <http://www.cherrycorp.com/english/sensors/sensorfaqs.htm>. Accessed 10/23/07.
- 12.2. Cherry. 2007. Speed and Proximity Sensors Catalogue. Online at <http://www.cherrycorp.com/english/sensors/pdf/SensorCatalog.pdf>. Accessed 11/14/07.
- 12.3. Measurement Computing Corporation. 2007. USB 4303 9513-based Counter/Timer User's Guide. Online at <http://www.measurementcomputing.com/PDFManuals/usb-4303.pdf>. Accessed 11/14/07.
- 12.4. SOP B1. 2006. Data Acquisition and Control Hardware. Standard Operating Procedure B1. Purdue Ag Air Quality Lab.
- 12.5. SOP B2. 2006. Data Acquisition and Control Software (AirDAC). Standard Operating Procedure B2. Purdue Ag Air Quality Lab.
- 12.6. SOP B3. 2006. AirDAC Raw Data Inspection System (ARDI). Standard Operating Procedure B3. Purdue Ag Air Quality Lab.
- 12.7. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 12.8. SOP B6. 2006. Data Processing Software (CAPECAB). Standard Operating Procedure B6. Purdue Ag Air Quality Lab.

DATA ACQUISITION AND CONTROL HARDWARE
Standard Operating Procedure (SOP) B1

DATA ACQUISITION AND CONTROL HARDWARE

Standard Operating Procedure (SOP) B1

Prepared by

Ji-Qin Ni and Bill W. Bogan

Reviewed by

Matt J. Darr (Ohio State Univ.), Teng-Teoh Lim and Albert J. Heber

Effective Date: December 28, 2007

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability.....	3
2. Summary of Method.....	3
3. Definitions.....	3
4. Health and Safety.....	4
5. Cautions.....	4
6. Interferences.....	5
7. Personnel Qualifications.....	5
8. Equipment and Supplies.....	6
9. Procedures.....	6
10. Data and Records Management.....	14
11. Quality Control and Quality Assurance.....	14
12. References.....	17
13. Contact Information.....	18

1. Scope and Applicability

- 1.1. Monitoring emissions from animal feeding operation (AFO) facilities requires a large number of sensors, instruments and sampling points. A system is needed to control all of these, and to collect the data which they generate.
- 1.2. The data acquisition and control (DAC) hardware will be used to take measurements and log digital and analog data, actuate controls of devices and control systems, and communicate with remote computers via an Ethernet line.
- 1.3. This SOP applies only to the specific hardware components involved in data acquisition and system control. Details on the individual instruments and devices which feed data to, and are under the control of, the DAC system, are provided in the SOPs for those individual instruments.

2. Summary of Method

This SOP describes the data acquisition and control (DAC) hardware for monitoring AFO emissions. It outlines the installation, configuration, and testing of individual DAC components or modules. Various Quality Control (QC) topics relevant to DAC hardware are also discussed.

DAC hardware that is used in the NAEMS for air emissions monitoring include FieldPoint modules (analog input from gas analyzers, thermocouple signals, and digital output) by NI (National Instruments Corporation, Austin, TX), and USB devices by MCC (Measurement Computing Corporation, Middleboro, MA). A PC with Windows XP running the AirDAC software (SOP B2) interfaces automatically with the DAC hardware to log data from various instruments and sensors, and to control designated devices. Devices that are controlled by the DAC system include solenoids in the Gas Sampling System (GSS) (SOP G1) and relays for heater controls in the heated raceways (SOP U3).

3. Definitions

- | | | |
|-------|-------|--|
| 3.1. | AFO | Animal feeding operation |
| 3.2. | AI | Analog input |
| 3.3. | DAC | Data acquisition and control |
| 3.4. | DI | Digital input |
| 3.5. | DO | Digital output |
| 3.6. | FP | FieldPoint |
| 3.7. | GSS | Gas sampling system |
| 3.8. | I/O | Input/output |
| 3.9. | MCC | Measurement Computing Corporation |
| 3.10. | NAEMS | National Air Emission Monitoring Study |
| 3.11. | NI | National Instruments Corporation |
| 3.12. | OFIS | On-farm instrument shelter |
| 3.13. | PAAQL | Purdue Agricultural Air Quality Lab |
| 3.14. | PC | Personal computer |

3.15. QC	Quality control
3.16. RH/T	Relative humidity/temperature
3.17. TC	Thermocouple
3.18. TEOM	Tapered element oscillating microbalance
3.19. USB	Universal serial bus

4. Health and Safety

- 4.1. Make sure that all wiring meets applicable electrical codes, and exercise the following precautions:
 - 4.1.1. Insulate signal connections for the maximum voltage for which the respective FieldPoint module is rated.
 - 4.1.2. Do not exceed the maximum voltage for which a particular module is rated.
 - 4.1.3. Do not wire a “live” module that is currently receiving electrical signals.
 - 4.1.4. Do not remove or add terminal bases when power is connected to the FieldPoint system.
 - 4.1.5. Avoid all direct contact with the terminal base signal wiring when “hot swapping” modules.
 - 4.1.6. Do not operate the DAC system around flammable gases or fumes.

5. Cautions

- 5.1. The FieldPoint modules are intended for indoor use, with an operating temperature of 25°C to 55°C, and relative humidity of 10% to 90%, in non-condensing air.
- 5.2. Do not substitute parts or modify the FieldPoint modular system. Use the system only with the modules, accessories, and cables specified in the installation instructions. Failure to do so could create a hazardous situation and/or damage the system itself.
- 5.3. All FieldPoint modules have built-in safety protections, which may be compromised if the units are damaged in any way. Do not use damaged FieldPoint modules; return them to the manufacturer for repair or discard them.
- 5.4. Only operate the FieldPoint modules at or below Pollution Degree 2 (generally only nonconductive pollution, although temporary periods of conductive pollution can occur). Typical outdoor dust at agricultural facilities constitutes Pollution Degree 3. Only operate FieldPoint modules inside the OFIS (SOP U1), or inside an enclosure with suitable particulate filters installed.
- 5.5. Use only soft, nonmetallic brushes to clean FieldPoint modules, and ensure that the modules are completely dry and contaminant-free before returning to use.
- 5.6. When handling I/O boards, use a wrist-grounding strap or hold on to a grounded object prior to opening any anti-static shipping packaging. Touch the antistatic container to the computer chassis before removing the board from the container.
 - 5.6.1. Do not touch any of the exposed pins or circuit connections on the board.
- 5.7. Before disconnecting any instrument or sensor from the FP-AI terminal base, turn off the instrument/sensor to avoid short-circuiting the signal wires, which could damage the instrument/sensor.

- 5.8. If a sensor that is already powered, e.g. an activity sensor, has to be connected to the data acquisition hardware, connect the signal ground wire to the data acquisition ground terminal before connecting the signal voltage wire. Otherwise, excessive high voltages may be introduced to the data acquisition system.

6. Interferences

- 6.1. Static electricity can interfere with or damage the DAC hardware. Make sure that all signal wires are properly shielded and grounded to avoid this interference.
- 6.2. Heat dissipated from adjacent equipment, for example, power supplies, can generate thermal gradients to thermocouple junction terminals. The FP-TC-120 corrects gradient errors up to 0.2°C; this is equivalent to a heat dissipation of approximately 4 W. Take steps to ensure that no other equipment on the DAC panel (Figure 1) in the vicinity of the TC-120 module dissipates more heat than this. For example, do not place a heater below the DAC panel.

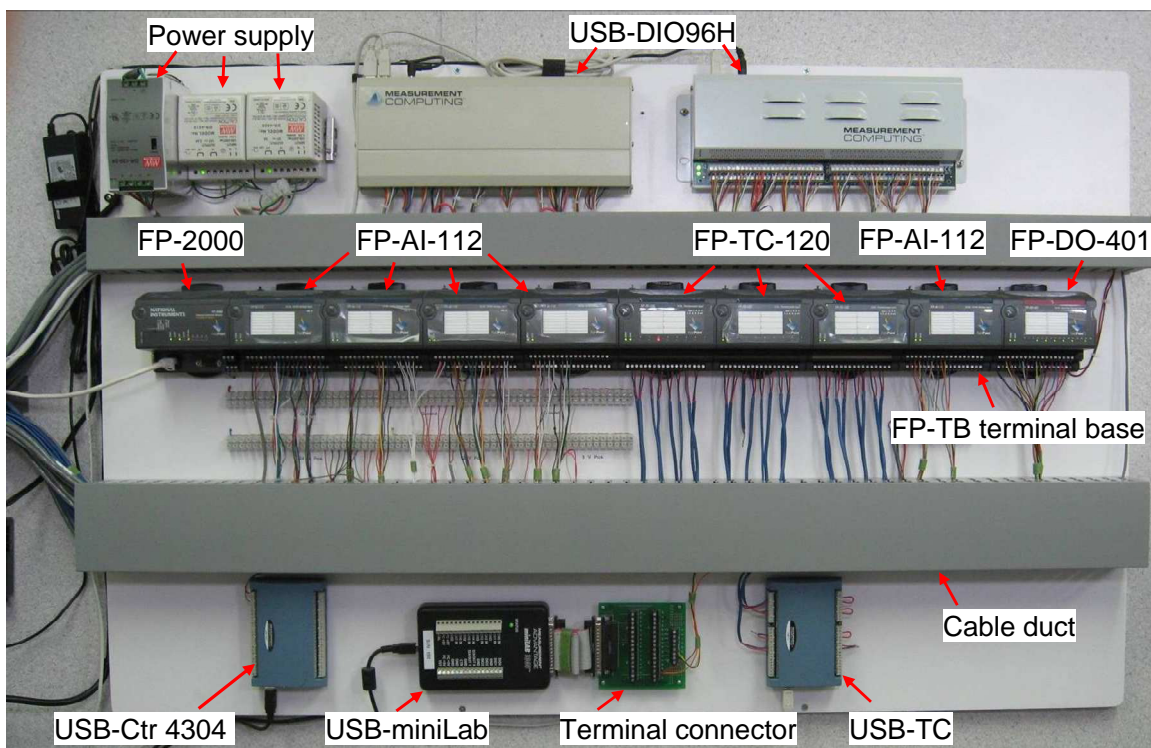


Figure 1. An example of DAC panel with power supply units, FieldPoint modules and USB devices.

7. Personnel Qualifications

- 7.1. Personnel should be trained in the use of the hardware before initiating any procedure.

- 7.2. Each analyst must read and understand the entire applicable instrument manual and this SOP before working with any of the individual DAC hardware components.
- 7.3. Personnel should have basic understanding of measurement and control systems.

8. Equipment and Supplies

- 8.1. FieldPoint modules (National Instruments Corporation, Austin, TX)
 - 8.1.1. FP-20xx FieldPoint Network Modules. One FP-20xx Network Module can support up to nine terminal bases.
 - 8.1.2. FP-AI-110 8-Channel, 16-Bit Analog Input Modules (optional)
 - 8.1.3. FP-AI-112 16-Channel, 16-Bit Analog Input Modules
 - 8.1.4. FP-TC-120 8-Channel Thermocouple Input Modules
 - 8.1.5. FP-DO-401 16-Channel, 5 to 30V Discrete Output Modules
 - 8.1.6. FP-TB-1 Terminal Bases (for FP-AI-11x and FP-DP-401 modules)
 - 8.1.7. FP-TB-3 Terminal Bases (for FP-TC-120 modules)
- 8.2. USB-based 96-bit digital I/O device with high current output (PCI-DIO96H, MCC, Middleboro, MA) (optional)
- 8.3. USB-based 10-channel counter device (USB-4303, MCC) (optional)
- 8.4. USB-based 8-channel 16-bit analog input device (USB-AI 608FS, MCC) (optional)
- 8.5. USB-based 8-channel thermocouple device (USB-TC, MCC) (optional)
- 8.6. USB-based 4-channel analog input (differential or 8-channel single-ended) and 28-channel DI device (USB miniLab, MCC) (optional)
- 8.7. Software
 - 8.7.1. LabVIEW (Version 7.1, NI), and Data Processing Report Generation Toolkit (Version 1.0.1, NI)
 - 8.7.2. FieldPoint™ for FieldPoint RT Controllers (Version 4.1 or later, NI)
 - 8.7.3. MCC DAQ Software (MCC), including InstaCal™ (Version 5.58 or later), TracerDAQ™ (Version 1.21 or later), and UL for LabVIEW.
- 8.8. Ethernet crossover cable - Category 5 (available in local store, e.g. RadioShack) for communication between FP network module and computer
- 8.9. USB cable(s) for connecting USB-based data acquisition devices to the computer

9. Procedures

- 9.1. Design the DAC hardware and software configuration by listing all the measurement and control channels in an Excel spreadsheet (Table 1).
 - 9.1.1. Arrange the FieldPoint module sequence as follows:
 - 9.1.2. FP-20xx (single) - FP-AI-11x (multiple) - FP-TC-120 (multiple) - FP-TC-410 (single or multiple). Except for the FP-20xx, the sequence of the other modules can be arranged differently.
 - 9.1.3. Arrange the instrument/sensor connection sequence to the analog inputs as follows:
 - 9.1.4. Gas analyzers - GSS sensors - TEOMs - Pressure transducers - Barn RH/T sensors - Weather sensors - Activity sensors - Other sensors.
- 9.2. Install LabVIEW (Version 7.1), Data Processing Report Generation Toolkit, FieldPoint,

and MCC DAQ software including InstaCal™, TracerDAQ™, and UL for LabVIEW.

9.3. Install and configure the FieldPoint modules.

9.3.1. Assemble individual DAC module bases and boards onto the DAC panel (Figure 1) in the OFIS. The FP modules may be dismounted when transporting the OFIS.

9.3.2. Mount the FieldPoint modules to the terminal bases (Figure 2) on the DAC Panel (also refer to FieldPoint Operating Instructions for details). Record all serial numbers of the modules prior to mounting.

9.3.3. Place a protective cover (included) over the unused bus connector of the last terminal base in the series.

9.3.4. Connect the RJ-45 Ethernet port of the FP-Network module to the PC using a category 5 Ethernet crossover cable.

Table 1. An example of a DAC measurement and control design table.

Data File, Sensor Arrangement, DAC Hardware, and Wire Code for IN2B (Nov. 1, 2007)																		
Data Col	File heading	DP	Sensor/controller	Sensor location	Monitoring/control location	Signal range	Measurement range	DAC hardware	Ch#	Gbit	DAC sig term#	DAC GND term#	To DAC sig	To DAC GND	To power V+	To power GND	Excitation power, VDC	
1	Date & time	---				PC clock		---	---	---								
2	Smpt loc#	0				LabVIEW		---	---	---								
3	Cal gas #	0	Environics	Rack		Log file		---	---	0								
4	Cal gas, ppm	2	Environics	Rack		Log file		---	---	1								
5	NH3, ppm	1	Innova	Rack	12 gas sampling locations	Serial port				2								
6	CO2, ppm	0	Innova	Rack	12 gas sampling locations	Serial port				3								
7	Reserved	0	Innova	Rack	12 gas sampling locations	Serial port				4								
8	Reserved	0	Innova	Rack	12 gas sampling locations	Serial port				5								
328	B9 cmvry, %d	0	Current switch	B9 Conveyor	B9 north manure conveyor			MiniLab1008	1	322	37		Black - Brown		Red - Red - #18		5	
329	B9 cmvry, %d	0	Current switch	B9 Conveyor	B9 south manure conveyor			MiniLab1008	2	323	36		Black - Blue		Red - Red - #18		5	
330	B9 cmvry, %d	0	Current switch	B9 Conveyor	B9 north manure conveyor			MiniLab1008	3	324	35		Black - Yel		Red - org - #18		5	
331	B9 cmvry, %d	0	Current switch	B9 Conveyor	B9 south manure conveyor			MiniLab1008	4	325	34		Black - blk		Red - red - #18		5	
332	Reserved	0						MiniLab1008	5	326	33							5
333	Reserved	0						MiniLab1008	6	327	32							5
334	Reserved	0						MiniLab1008	7	328	31							5
335			Solenoid #1	B9 Inlet	B9 air inlet at center and fan 33			FP-DO-401-1	0	1			Blk (Red Paired)					24
336			Solenoid #2	B9 Inlet	B9 air inlet at center and fan 33			FP-DO-401-1	1	2			Yellow (Blk Paired)					24
337			Solenoid #3	B9 Fans 21	B9 Fans 21 N and S			FP-DO-401-1	2	3			Red (Blk Paired)					24
338			Solenoid #4	B9 Fans 21	B9 Fans 21 N and S			FP-DO-401-1	3	4			Blk (Yellow Paired)					24
339			Solenoid #5	B9 Fans 28	B9 Fans 28 N and S			FP-DO-401-1	4	5			Grn (Red Paired)					24
340			Solenoid #6	B9 Fans 28	B9 Fans 28 N and S			FP-DO-401-1	5	6			Wht (Blk Paired)					24
341			Solenoid #7	B9 Fans 33	B9 Fans 33 N and S			FP-DO-401-1	6	7			Red (Grn Paired)					24
342			Solenoid #8	B9 Fans 33	B9 Fans 33 N and S			FP-DO-401-1	7	8			Blk (Wht Paired)					24
343			Solenoid #9	B9 N Belt	B9 N Belt exhaust			FP-DO-401-1	8	9			Yellow (Red Paired)					24
344			Solenoid #10	B9 S Belt	B9 S Belt exhaust			FP-DO-401-1	9	10			Grn (Blk Paired)					24
345			Solenoid #11	B9 S Belt	B9 S Belt exhaust			FP-DO-401-1	10	11			Red (Yellow Paired)					24
346			Solenoid #12	B9 S Belt	B9 S Belt exhaust			FP-DO-401-1	11	12			Blk (Grn Paired)					24
347			Solenoid #13	Manure shed	Manure shed E wall and Eave			FP-DO-401-1	12	13			Brown (Red Paired)					24
348			Cooling Fan	GSS				FP-DO-401-1	13	14			Blue (Blk Paired)					24
349			Raceway	Rwy B8				FP-DO-401-1	14	15								24
350			Raceway	Rwy B9				FP-DO-401-1	15	16								24

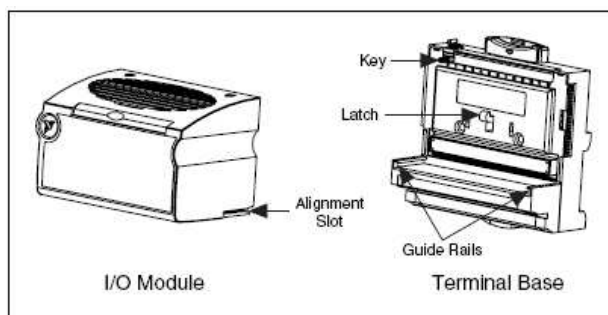


Figure 2. Connection points between I/O modules and terminal base.

- 9.4. Verify that the power cord and fuse connection to the power supplies are all correct and in good condition.
- 9.5. Plug in power supply. The FP-Network module will run a self-test. At the conclusion of this self-test (when the “STATUS” LED begins flashing) the module is ready for configuration (refer to FP-2000/2010/2015 User Manual, Part number 370705A-01 for details).
- 9.6. At initial setup, launch Measurement & Automation Explorer software in the DAC computer. On the Configuration panel, expand “Remote System” and follow the example illustrated from Figures 3 to 10.

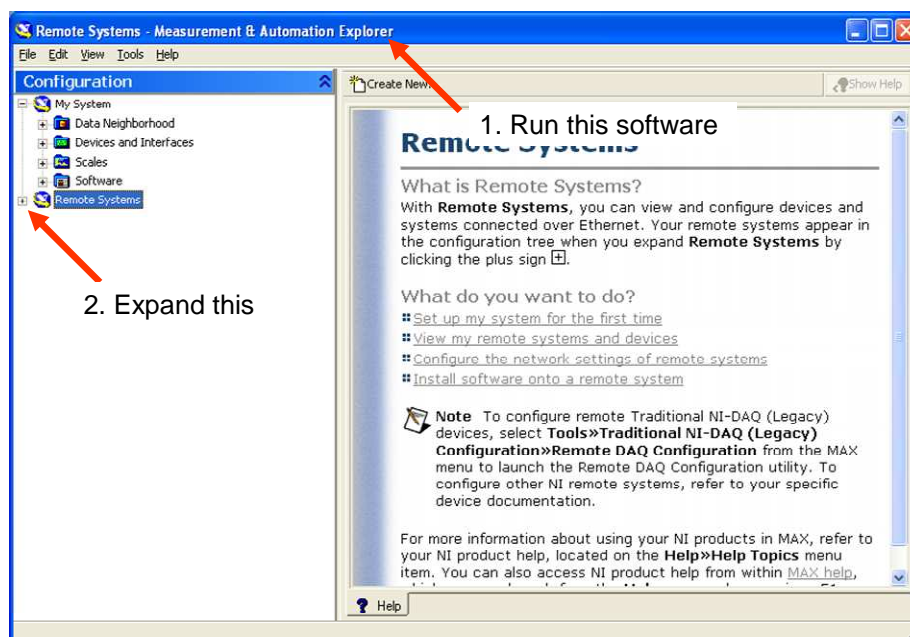


Figure 3. Configure the FieldPoint modules at Measurement & Automation Explorer.

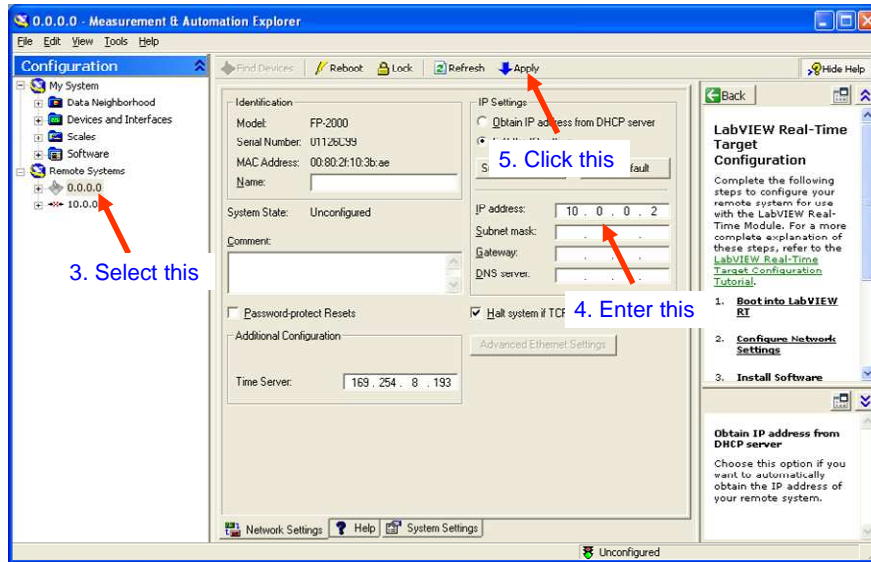


Figure 4. Configure the FieldPoint modules Step 3-5.

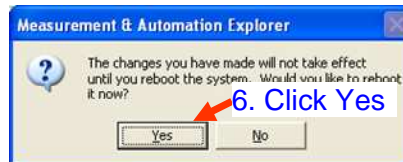


Figure 5. Configure the FieldPoint modules Step 6.

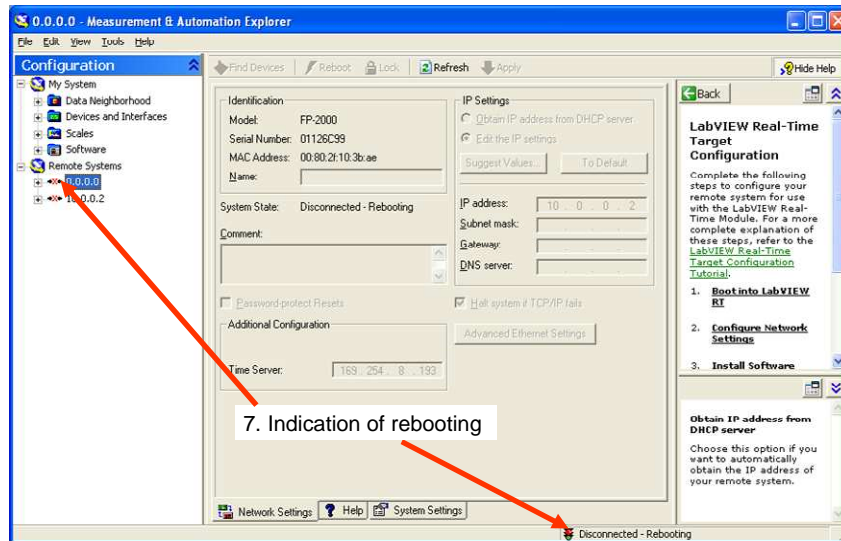


Figure 6. Configure the FieldPoint modules Step 7.

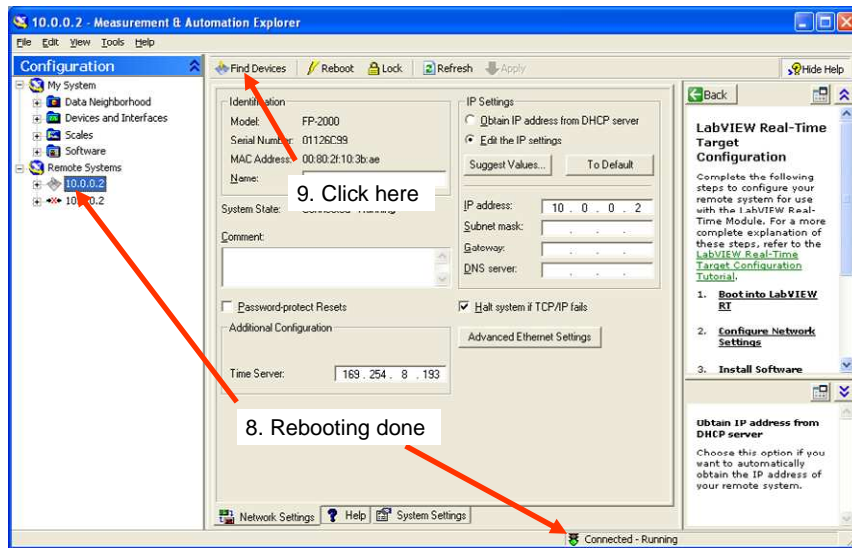


Figure 7. Configure the FieldPoint modules Step 8-9.

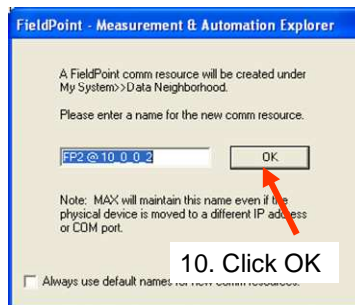


Figure 8. Configure the FieldPoint modules Step 10.

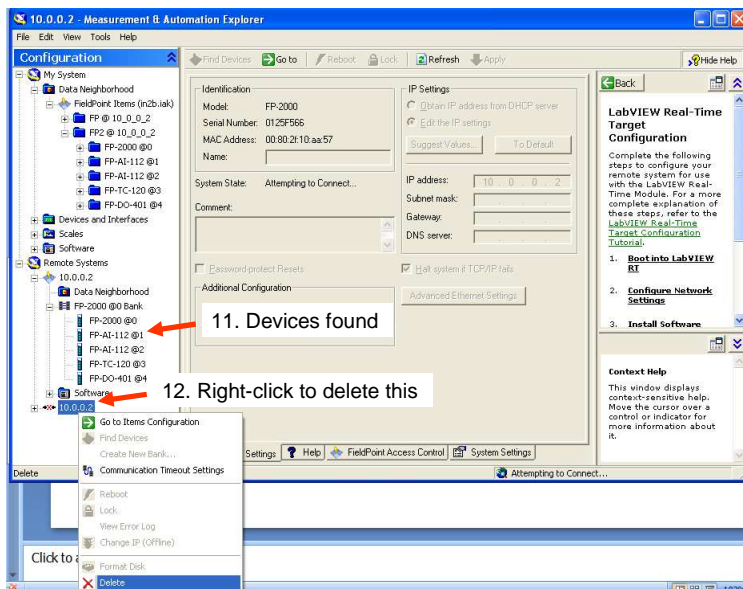


Figure 9. Configure the FieldPoint modules Step 11-12.

- 9.6.1. In the “Remote Systems” box, configure the “Range” of analog input channels one by one (Figure 10) according to the DAC design table (Table 1).

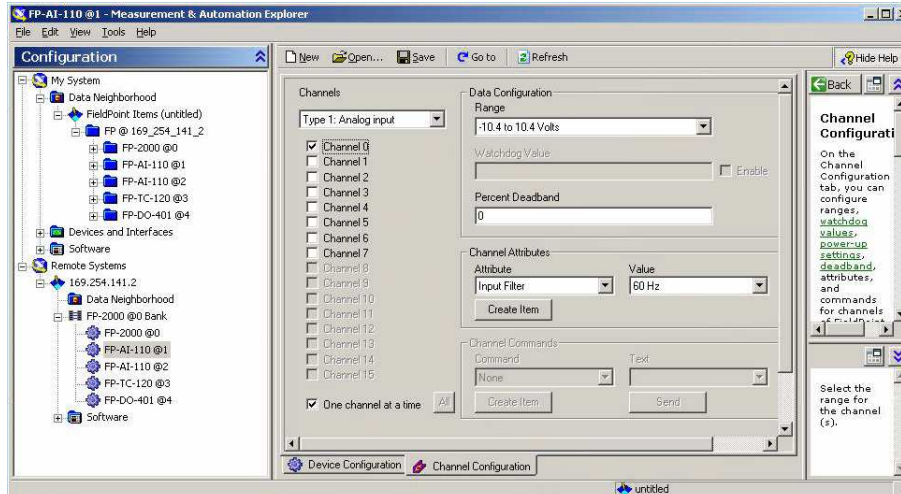


Figure 10. Configuring channels for analog FP-AI-11x modules.

- 9.6.2. Set “Range” at “-270 to 1770 Celsius” and “Channel Attributes” at “Thermocouple type T” for all channels in FP-TC-120 modules (Figure 11).

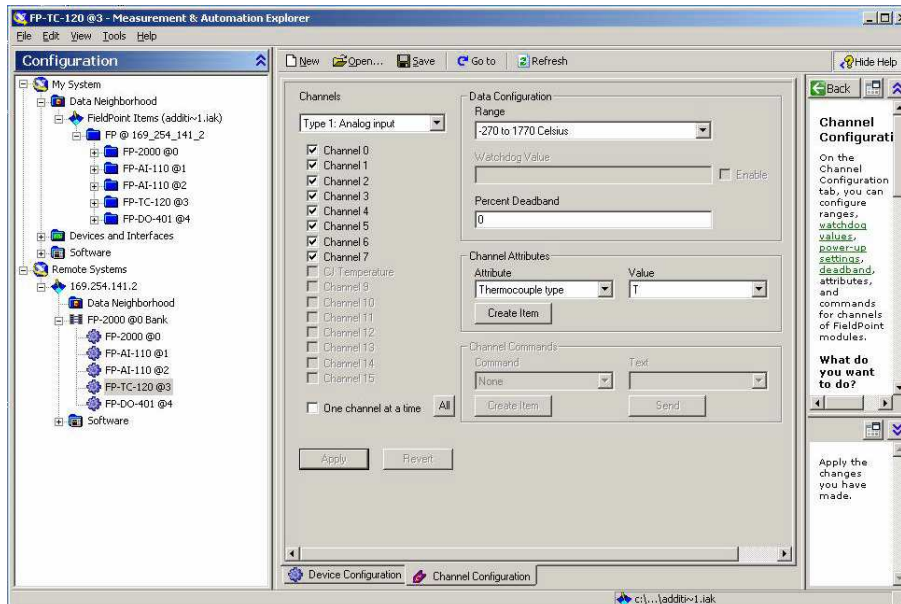



Figure 11. Configuring channels for thermocouple FP-TC-120 modules.

- 9.6.3. Test the hardware and configuration. In the “Data Neighborhood,” select the FieldPoint module. At the top of the right panel, click “Start”. If the “Status” column for the FP-AI-11x, FP-TC-120, and FP-DO-401 modules indicates “Successful” for all channels, the modules are working properly (Figure 12). If “Successful” is not

indicated for all channels, refer to the “Help” section of the Measurement & Automation Explorer to debug. Contact the manufacturer if necessary (Section 13.1).

- 9.6.4. Save the configuration by clicking  and assigning the filename C:\AirDAC\Program\ XXnY.iak, where XXnY is the abbreviation of the site.

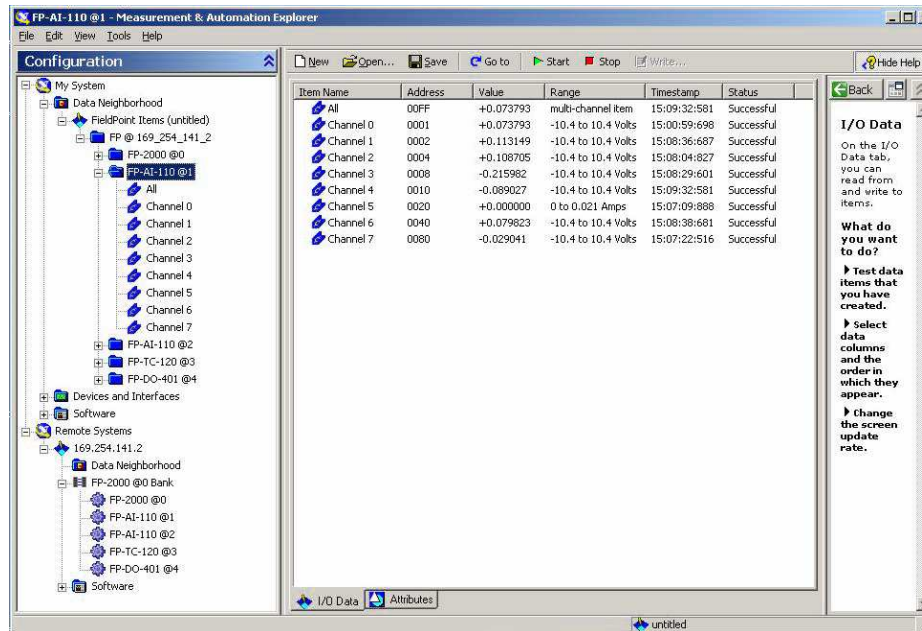


Figure 12. A test of data acquisition signals in Measurement & Automation Explorer.

- 9.6.5. Refer to “Help” in Measurement & Automation Explorer if further assistance is required during the configuration process.
- 9.7. Install and configure the USB-based data acquisition devices, e.g. the DIO96H device.
- 9.7.1. Connect the USB-DIO 96H to one of the PC’s USB ports with a USB cable.
- 9.7.2. Connect the AC Adapter to the power and the USB-DIO 96H.
- 9.7.3. A “Found New Hardware” balloon will appear on the PC. Another balloon will appear when the device is successfully installed.
- 9.7.4. Run InstaCal from the desktop or from Start->Programs->InstaCal (Figure 13). If the hardware is successfully detected, a Plug and Play Board Detection window (Figure 14) will appear. Click the OK button to close the window.

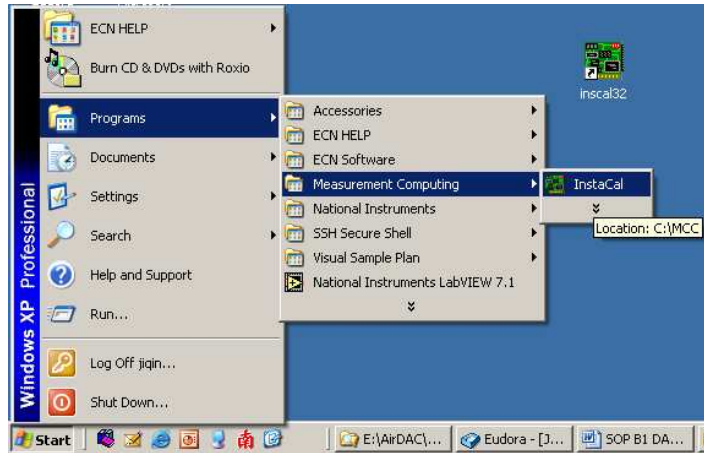


Figure 13. Running InstaCal, the MCC hardware configuration program.

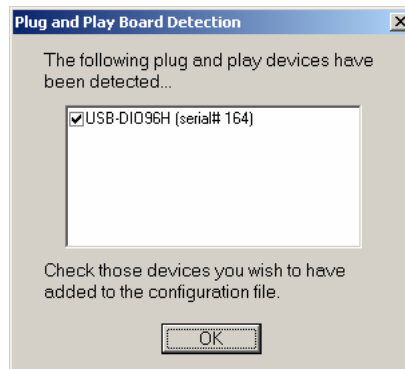



Figure 14. Detection of the USB-DIO 96H by InstaCal.

- 9.8. The device will appear under the Universal Serial Bus heading in InstaCal (Figure 15). Record the board number (Board# n), as this will be used in the AirDAC program (SOP B2). Close InstaCal by clicking the  or selecting File > Exit.

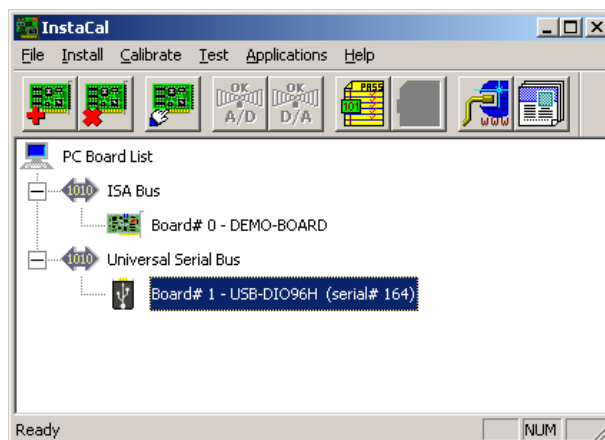


Figure 15. USB DIO96H device configured by InstaCal.

- 9.9. Connect measurement/control device signals to DAC hardware.
- 9.9.1. Turn off the power to the DAC hardware, gas analyzers, TEOMs and other sensors before initial connection to avoid potential damage of the devices.
- 9.9.2. Connect analog input signals to the FP-AI terminal bases according to the DAC channel table (Table 1).
- 9.9.3. Connect thermocouple wires to the FP-TC terminal bases. On Type T thermocouples, the blue (copper) is + and the red (constantan) is -. If shielded extension wires are used, connect one end of the shield to the corresponding "COM" terminal as per the table below.

Input Channel	Terminal Numbers		
	IN(+)	IN(-)	COM
0	1	2	18
1	3	4	20
2	5	6	22
3	7	8	24
4	9	10	26
5	11	12	28
6	13	14	30
7	15	16	32

- 9.9.4. Connect DI signals to the USB DIO96H device connection terminals according to the DAC channel table.
- 9.9.5. Connect DO for controlling devices (GSS solenoids, raceway heating relays) to the FP-DO terminal bases according to the DAC channel table.
- 9.9.6. Connect other signal wires to the relevant DAC devices, e.g. USB-based counters if they are configured in the system.

10. Data and Records Management

- 10.1. Maintain all laboratory records in a worksheet of the designated electronic field notes spreadsheet. Supplement this electronic record with a bound record book, which should also contain bound copies of the electronic record.
- 10.2. Manage all data and files (including the configuration files) according to SOP B5.
- 10.3. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes. Overstrike all errors in writing with a single line. Initial and date all such corrections.

11. Quality Control and Quality Assurance

- 11.1. Calibration of FieldPoint modules
- 11.1.1. National Instruments products require calibration two years after their manufacture, and every year thereafter. Calibration of FP-AI modules involves expensive

- instrumentation, and is preferably done by returning the module to the manufacturer.
- 11.1.2. Maintain calibration records in a separate sheet in the Excel file with the DAC measurement and control table (Table 1). Place a calibration sticker on each piece of hardware, indicating date of last calibration.
 - 11.2. Test the FP-AI modules at the initial setup and every year thereafter.
 - 11.2.1. Disconnect the instrument/sensor signal from the channel to be tested
 - 11.2.1.1 Before disconnecting from the FP-AI terminal base, turn the instrument/sensor off to avoid short-circuiting the signal wires.
 - 11.2.2. Use a short wire to short-circuit the two terminals that were connected to the instrument/sensor.
 - 11.3. Launch Measurement & Automation Explorer (Section 9.6.3) and check the values displayed in the right panel (Figure 12). The channel being tested should display a stabilized, near-zero value ($< \pm 1$ mV or $< \pm 1$ μ A). If it is not stable, the channel is not working properly.
 - 11.4. Disconnect the short-circuit wire from the terminals. Connect a known voltage or a known current to the terminals. Use a voltage or current equivalent to the high range signal of the instrument/sensor that is hooked up to the channel. For example, use 10.000 V to test the channel for a gas analyzer that has signal range from 0 to 10 VDC. Check the value of the channel in the Measurement & Automation Explorer. The displayed value should stabilize close to the generated value (within $\pm 0.1\%$). If it is not stable, the channel is not working properly.
 - 11.5. If the displayed values from the zero test are $> \pm 1$ mV or $> \pm 1$ μ A (Sections 11.2.2-11.3) or are $> \pm 0.1\%$ compared with the known values from the voltage/current test (Section 11.4), but are stable, use the AirDAC program to make corrections by entering the zero and voltage/current values in “3. Signal low” and “4. Signal high,” respectively, in the AirDAC DAQ table (SOP B2).
 - 11.6. Do not use any channel that fails the voltage test. If a channel fails the current signal test, test it for voltage signals.
 - 11.6.1. A channel that works properly with voltage signals but not current signals may be used for voltage signal acquisition.
 - 11.6.2. Re-arrange the channels when necessary by switching the channels and adjusting the “10. Global ch#” in the AirDAC DAQ table (SOP B2) to avoid using any non-working channels.
 - 11.6.3. If the module does not have enough functional channels, replace it and send it to the manufacturer for repair.
 - 11.7. Test the FP-TC modules at setup and every year thereafter. Combine the FP-TC channel test with thermocouple calibration (SOP E1). Do not use any channel that fails the test.
 - 11.8. Verifying and testing the FP-DO-401 channels
 - 11.8.1. Verify DO channels that are used to control the GSS solenoids as part of the daily data check. If the air sample flow rate at a certain sampling location decreases by more than 30% of the initial value during the 10-min sampling time, the relevant solenoid in the GSS may have been receiving decreasing energizing power, which can be due to a mal-functioning FP-DO-401 channel. To test this, measure the voltage that the solenoid receives (while it is being energized) using a multimeter. If the voltage is not stable, the FP-DO-401 relevant channel should be removed from use.

11.9. Test of USB DIO96H digital input signals

11.9.1. Launch InstaCal and select the USB DIO96H device. In the “Test” menu, select “Digital” (Figure 16). A new test window will appear.

11.9.2. Select the Internal DIO test tab and click Test (**Error! Reference source not found.**).

11.9.3. If it is necessary, select the External DIO test tab and follow the instructions to the external signal test.

11.9.4. Make sure all DI channels that are being used are working properly to acquire data.

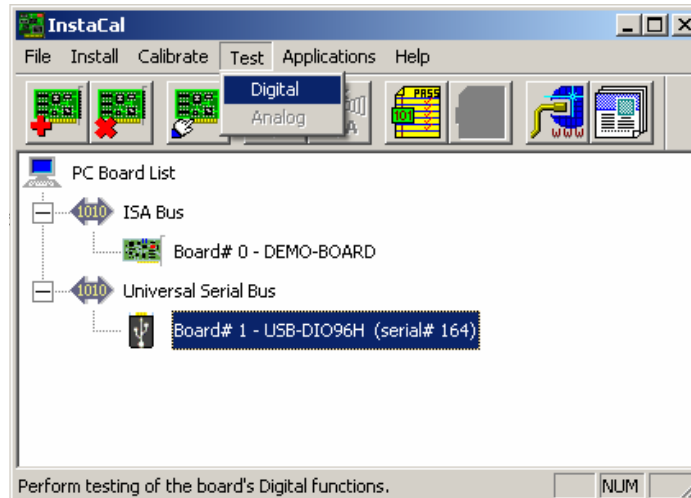


Figure 16. Test the USB DIO96H device using InstaCal.

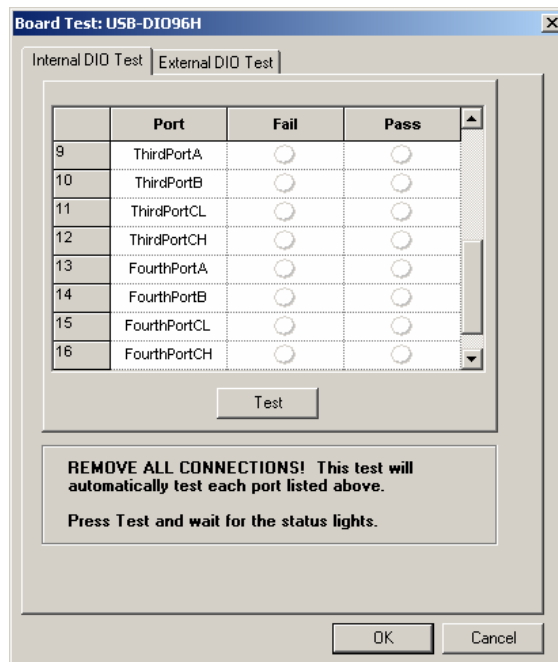


Figure 17. Internal DIO test tab in InstaCal.

11.10. Test of USB-4303 counter signals

11.10.1. Run the “MCC USB-4303 Event Counter Tester.vi” located in C:\AirDAC\Program\NI Special\ by double-clicking it.

11.10.2. In the window that appear (Figure 18), enter the USB 4303 device board number in the Board# on the left side of the window. The Event Count, Fan RPM and Error Message will indicate the status of the hardware and the signals that each channel receives.

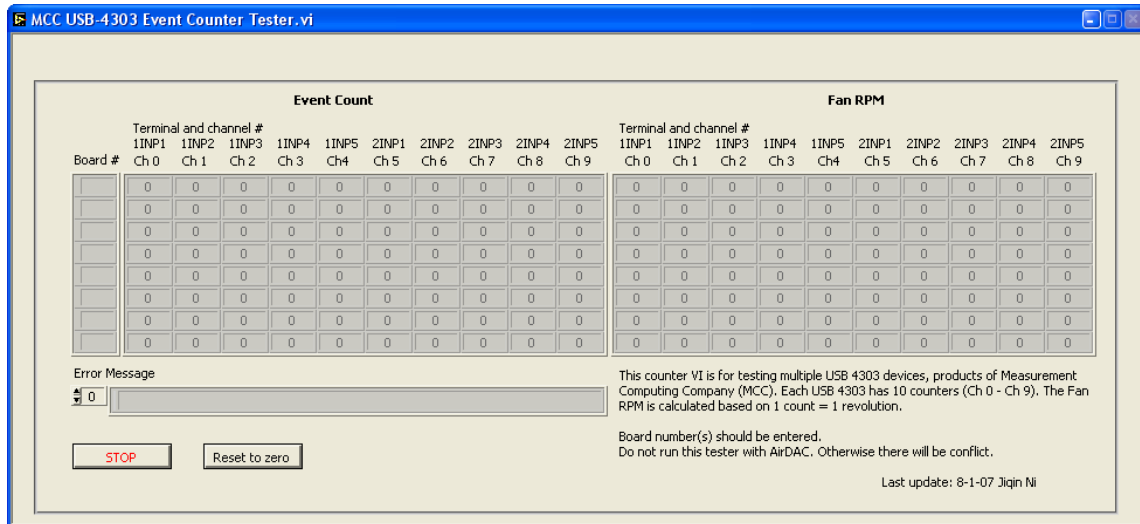


Figure 18. Test the USB 4303 counter device.

12. References

- 12.1. AI Module Manual. 2002. FieldPoint Operating Instructions: FP-AI-110 and cFP-AI-110. NI, Austin, TX.
- 12.2. AI Module Manual. 2004. FP-AI-112 and FP-AI-112 Operating Instructions. NI, Austin, TX.
- 12.3. DO Module Manual. 2002. FieldPoint Operating Instructions: FP-DO-401 and cFP-DO-401. NI, Austin, TX.
- 12.4. TC Module Manual. 2002. FieldPoint Operating Instructions: FP-TC-120 and cFP-TC-120. Part Number: 323344A-01. NI, Austin, TX.
- 12.5. FP User Manual. 2003. FP-2000/2010/2015 User Manual. NI, Austin, TX.
- 12.6. Measurement Computing. 2006. USB-DIO 96H/50 User's Guide. File location: C:\Program Files\Measurement Computing\DAQ\Documents\UsersGuides\USB\USB-DIO96H.pdf.
- 12.7. Measurement Computing. 2006. USB-4303 User's Guide. File location: C:\Program Files\Measurement Computing\DAQ\Documents\UsersGuides\USB\USB-4303.pdf.
- 12.8. Measurement Computing. 2006. USB-1608FS User's Guide. File location: C:\Program Files\Measurement Computing\DAQ\Documents\UsersGuides\USB\USB-1608FS.pdf.
- 12.9. Measurement Computing. 2006. USB-TC User's Guide. File location: C:\Program Files\Measurement Computing\DAQ\Documents\UsersGuides\USB\USB-TC.pdf.

- 12.10. Measurement Computing. 2006. MiniLAB-1008 User's Guide. File location:
C:\Program Files\Measurement Computing\DAQ\Documents\UsersGuides\
USB\MiniLAB-1008.pdf.
- 12.11. Quick Start Guide - Measurement Computing Data Acquisition. September 2005. MCC,
Middleboro, MA.
- 12.12. SOP B2. 2006. Data Acquisition and Control Software (AirDAC). Standard Operating
Procedure B2. Purdue Ag Air Quality Lab.
- 12.13. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue
Ag Air Quality Lab.
- 12.14. SOP E1. 2006. Temperature Measurement Using Thermocouples. Standard Operating
Procedure E1. Purdue Ag Air Quality Lab.
- 12.15. SOP G1. 2006. The PAAQL Gas Sampling System. Standard Operating Procedure G1.
Purdue Ag Air Quality Lab.
- 12.16. SOP U1. 2006. On-farm Instrument Shelters for Barn Sources. Standard Operating
Procedure U1. Purdue Ag Air Quality Lab.
- 12.17. SOP U3. 2006. Heated Raceway. Standard Operating Procedure U3. Purdue Ag Air
Quality Lab.

13. Contact Information

13.1. Manufacturers:

13.1.1. National Instruments Corporation

13.1.2. 11500 N Mopac Expwy

13.1.3. Austin, TX 78759-3504.

13.1.4. Technical Support: (800) 531-5066

13.1.5. Measurement Computing Corporation

16 Commerce Boulevard

Middleboro, MA 02346

(508) 946-5100

info@measurementcomputing.com

13.1.6. Technical Support: measurementcomputing.com/contact.html

13.2. PAAQL: odor@purdue.edu, heber@purdue.edu, <http://www.AgAirQuality.com>.

DATA ACQUISITION AND CONTROL SOFTWARE (AirDAC)

Standard Operating Procedure (SOP) B2

**DATA ACQUISITION AND CONTROL SOFTWARE (AirDAC)
Standard Operating Procedure (SOP) B2**

Prepared by

Ji-Qin Ni and Bill W. Bogan

Reviewed by

Teng-Teoh Lim and Albert J. Heber

Effective Date: November 6, 2006

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions	5
4. Interferences	5
5. Personnel Qualifications	6
6. Equipment and Supplies	7
7. Procedures.....	7
8. Data and Records Management.....	14
9. Quality Control and Quality Assurance.....	14
10. References	15
11. Contact Information.....	16

1. Scope and Applicability

- 1.1. Specialized software is required for Data Acquisition and Control (DAC) in the National Air Emission Monitoring Studies at livestock facilities. This SOP describes the operating procedure for the specialized DAC software, AirDAC.
 - 1.1.1. This software acquires data from an array of sensors and analyzers, of which the analog or digital signal cables are connected to the DAC hardware described in SOP B1.
 - 1.1.2. The software converts the incoming analog data signals into data with engineering units, based on the signal range and measurement range settings. It allows further adjustments to be made to the data by applying linear equations, which can be based on calibration models. It averages the digital signals over a pre-specified range of time.
 - 1.1.3. The software provides digital output controls for various devices, including, for example, the solenoids in the Gas Sampling System (GSS).
- 1.2. The software acquires up to 254 analog and digital signals at a sampling frequency of 1 Hz. The software controls up to 32 digital outputs for controlling the GSS, raceway heaters, and other devices. The software averages data every 15 and 60 s, and saves them into two separate data files.
- 1.3. The data collected by AirDAC are analyzed according to SOPs B3, B4, and B6.

2. Summary of Method

The AirDAC software, developed by PAAQL in the LabVIEW programming language (National Instruments Corporation, Austin, TX), works with data acquisition and control hardware (National Instruments and Measurement Computing Corporation). The software controls sampling location, acquires and displays real-time data, delivers data files, and displays the system's operating status to research personnel. Figure 1 provides a flow diagram for AirDAC.

AirDAC accepts initial configuration at startup. Two data files, for 15-s and 60-s data records, respectively, are created at the initial startup, and again at midnight of each subsequent day. The software reads DAQ, digital outputs (DO), alarm, and email configurations before it reads data from DAQ hardware every second in the main (DAC) loop. The acquired data are calculated using parameters and coefficients defined in the configuration, and averaged and saved every 15 s and 60 s into two separate data files.

The second loop, parallel to the DAC loop, displays real-time monitoring and control data, checks the measurement system, sends alarm and data emails, and accepts dynamic run-time re-configuration. The run-time configuration accepts sensor and measurement range changes, linear models for data correction (e.g. calibration curves), alarm and email settings, DAQ channel changes, DO settings, and sampling sequence and sampling cycle (sampling time and frequency) changes. Whenever a new configuration is applied, AirDAC automatically saves it in an Excel file.

AirDAC uses All-data Monitoring, visual alarm display, alarm emails, and 1-min data emails to ensure measurement quality control. AirDAC also allows automatic and manual controls over air sampling locations.

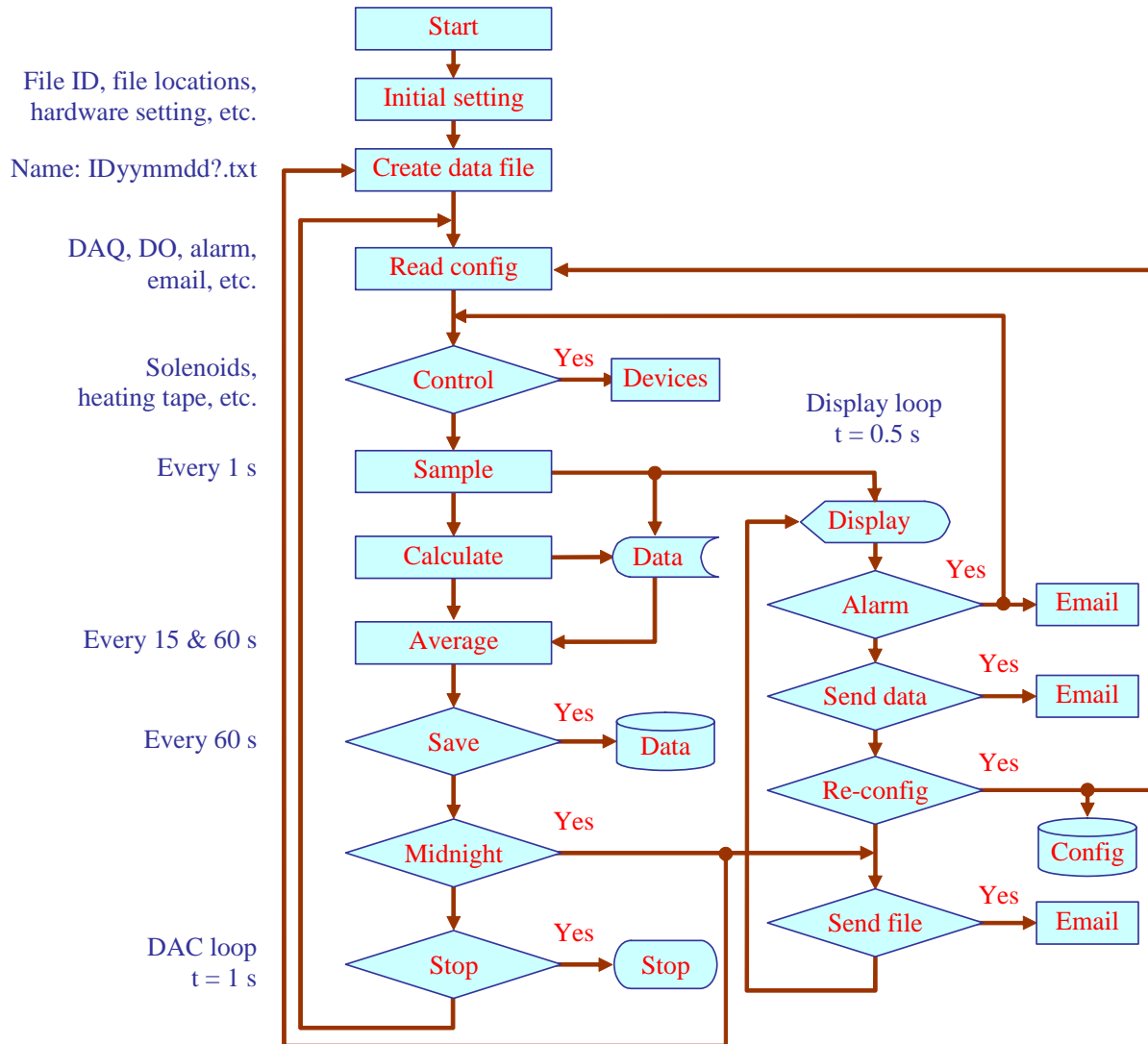


Figure 1. Flow diagram of AirDAC software.

3. Definitions

3.1. AI	Analog input
3.2. AirDAC	Air emission monitoring Data Acquisition and Control software, written in the LabVIEW programming language
3.3. CPU	Central processing unit of a PC
3.4. DAC	Data acquisition and control
3.5. DAQ	Data acquisition
3.6. Data reading	One data point read by AirDAC (i.e. every second).
3.7. Data record	A single data point saved in data files by AirDAC. It is the average of either 15 or 60 1-s data readings.
3.8. DIO	Digital input/output
3.9. DO	Digital output
3.10. GSS	Gas Sampling System (SOP G1)
3.11. LabVIEW	Laboratory Virtual Instrumentation Engineering Workbench. A platform and development environment for a visual programming language (a dataflow language), produced by NI
3.12. MCC	Measurement Computing Corporation
3.13. NI	National Instruments Corporation
3.14. PAAQL	Purdue Agricultural Air Quality Laboratory
3.15. S	The signal output currently being received from an instrument
3.16. Sampling cycle	The amount of time that the sampling system takes to scan through all the sampling locations, and return to the starting location. For example, the sampling cycle is 2 h (120 min) for a monitoring site with 12 locations, with a sampling time of 10 min each.
3.17. Sampling frequency	The frequency of sampling at a particular sampling location relative to the sampling cycle. For example, when the ambient sample location is set at a sampling frequency of 1/3, it is sampled once every three sampling cycles.
3.18. Sampling period	Duration of time used to sample at a certain location. For example, when the ambient sampling time is set at 20 min, it is sampled for 20 min before the sampling system switches to the next location.
3.19. Sampling sequence	The sequence of sampling among different sample locations
3.20. TC	Thermocouple
3.21. USB	Universal serial bus
3.22. X	The data converted from analog signals into engineering units based on sensor signal range and measurement range settings, or the data averaged from digital signals into percentage of time.
3.23. Y	The adjusted X values after applying linear equations, which can be calibration curves. These are the data saved in data files.

4. Interferences

- 4.1. LabVIEW might be interfered with by other running software. One such interference was caused by the Purdue University wireless internet connection, Purdue Air Link. Other similar software may cause similar interferences. No solution, other than to avoid running other software while AirDAC is running, has been found.

- 4.2. Data reading may slow to more than 1 s if other major software is running and using a large percentage of the PC's CPU capacity. Close other software if the DAC loop time, displayed in the AirDAC front panel's M/C tab, shows an average of more than 1.000 s.
- 4.3. Electric static charges (e.g. from the operation of a >20 kV electrostatic space charge system for PM removal) may interfere with DAC hardware, DAC software, and computer operations. These static charges could also damage the DAC hardware. Properly shield the sensor signal wires, and ground the shield.
- 4.4. After the AirDAC startup, during which the configuration file is read, Excel will not show the "Save changes?" warning dialog (**Figure 2**) when an Excel workbook that has unsaved changes is being closed. Save open and unsaved Excel workbooks, close them, and close Excel. The problem will be solved when Excel is re-opened.

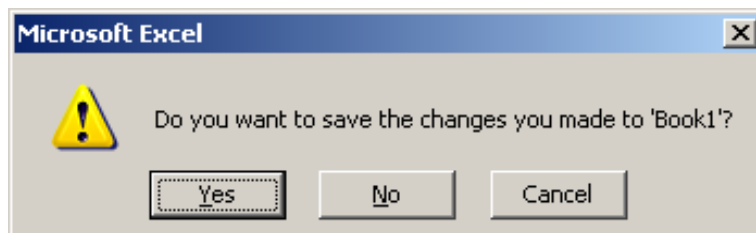


Figure 2. The Excel "Save changes" warning dialog box.

- 4.5. The number of data files automatically saved by AirDAC is limited to 27 per day, because the last character "?" in the data file names (IDsyymmdd?.txt and IDyyymmdd?.txt) will become "|", and a Windows file name cannot contain this character. However, this only happens during AirDAC testing (during which the software may be restarted this many times in one day), and not during normal use.
 - 4.5.1. If this situation happens, delete or remove some of the IDsyymmdd?.txt and IDyyymmdd?.txt files for the day from the "Data" folder to restart AirDAC.
- 4.6. The LabVIEW email feature has been found to not work with some computers. More information about this can be found in the web page: <http://digital.ni.com/public.nsf/allkb/42556976302AFF3C86256D7C0080AB5C>). This may be related to the installation of Microsoft Windows XP Service Pack 2 (SP2). More information can be found in: <http://support.microsoft.com/default.aspx?kbid=842242>.



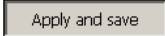
5. Personnel Qualifications

- 5.1. Most of these procedures assume a familiarity with the use of personal computers, general computer application software (e.g. MS Windows, MS Office), and a basic understanding of relevant data acquisition software, including Measurement & Automation Explorer and LabVIEW software.
- 5.2. LabVIEW Self-Paced Training (<http://sine.ni.com/nips/cds/view/p/lang/en/nid/10450>) or LabVIEW Basics I: Introduction (<http://sine.ni.com/nips/cds/view/p/lang/en/nid/2241>) is recommended.
- 5.3. All personnel operating AirDAC are required to read and understand this SOP.
- 5.4. If the DAC hardware includes MCC USB DIO devices, the operator is also required to read the instructions of those devices.
- 5.5. Only authorized personnel are allowed to work on AirDAC, or modify its configuration.

6. Equipment and Supplies

- 6.1. PC with 1.5 GHz or higher CPU speed, 512 MB or more RAM, and 80 GB or more hard disk space
- 6.2. Software
 - 6.2.1. Windows XP operating system
 - 6.2.2. LabVIEW version 7.1 (NI) and LabVIEW Report Generation Toolkit (NI)
 - 6.2.3. FieldPoint™ for FieldPoint RT Controllers (Version 4.1 or later) (NI)
 - 6.2.4. MCC DAQ Software (MCC), including InstaCal™ (Version 5.58 or later), TracerDAQ™ (Version 1.21 or later), and UL for LabVIEW
 - 6.2.5. AirDAC software, stored in C:\YYsiten\AirDAC\Program\
 - 6.2.6. AirDAQ Configuration file, stored in C:\YYsiten\AirDAC\Program\
- 6.3. FieldPoint modules (NI) connected to the computer and configured (SOP B1)
- 6.4. USB-based 96-bit digital I/O device with high current output (PCI-DIO96H, MCC) connected to the computer and configured

7. Procedures

- 7.1. Prepare AirDAQ configuration file
 - 7.1.1. Make a copy of the “AirDAC Config template.xls” file (**Figure 3**), provided by PAAQL, to C:\YYsiten\AirDAC\Program\. Rename it as “AirDAC Config YYn.xls”, where YY is the two-letter state abbreviation and n is a site-specific number.
 - 7.1.2. Modify the DataConfig, DOconfig, and Email sheets according to the Notes in the sheets, and modify the DAC design table according to SOP B1. Alternatively, these can be configured while running AirDAC (See Sections 7.3.9 – 7.3.17).
- 7.2. Test DAC hardware
 - 7.2.1. Locate the C:\YYsiten\AirDAC\Program\YYsiten.iak file in Windows Explorer, and double-click to run. Or run the Measurement & Automation Explorer from Windows Start->Programs->National Instrument->Measurement & Automation (Figure 4), then open the iak file from C:\YYsiten\AirDAC\Program\YYsiten.iak file. The iak file was generated during DAC hardware setup (See SOP B1 for details).
 - 7.2.2. Check each FieldPoint module to ensure that it is configured correctly, and that it is ready to acquire data.
 - 7.2.3. If modifications are made and need to be kept, save the YYsiten.iak file.
- 7.3. Test and configure the AirDAC: Initial operation
 - 7.3.1. Run the C:\YYsiten\AirDAC\Program\AirDAC start.vi file by double-clicking it. An AirDAC start window (Figure 5) will open.
 - 7.3.2. Click  to modify initial configurations. The  button becomes . In the “Initial setting” tab (Figure 5) enter a 2-letter data file ID.
 - 7.3.3. Check the “Wind direction” if wind direction measurement is included. Enter the wind direction column number, in which the wind direction data is saved in raw data files. Select the wind direction calculation method between “Scalar” (Webmet, 2005) and “Take last second” only. Enter the WD in degrees to correct wind sensor installation error. Measure the error from due north. For example, if the wind direction sensor shows due east when it should be due north, the error is 90°.

The screenshot shows an Excel spreadsheet titled "AirDAC Config template.xls". The spreadsheet contains a table with 15 data columns and various parameters. The table is as follows:

Data column #	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	60
1. Data heading	Date & time	Smpl loc#	NH3_ppm	H2S_ppb	SO2_ppb	CO2_ppm	+3 MSA_pp	Smpl P_Pa	P smpl ex	F5mpl O_L/m	Smpl RH, %	Smpl T, C	AI NOx_ppm	AI NO2_ppm	AI NH3_ppm	Aet2_VDC
2. Digit of prec.	0	3	3	3	3	3	3	2	2	2	1	1	3	3	3	2
3. Signal lo (SL)		0	0	0	0	0.004	0	0.004	0.004	0	0	0	0	0	0	2
4. Signal hi (SH)		10	10	10	0.02	10	0.02	0.02	0.02	5	1	1	10	10	10	3
5. Range lo (RL)		0	0	0	0	0	0	-34474	-37.35	0	0	-40	0	0	0	0
6. Range hi (RH)		50	10000	10000	10000	1000	1000	34474	37.35	10	100	60	50	50	50	1
7. Absolute (abs)		0	0	0	0	0	0	0	0	0	0	0	0	0	0	1
8. Slope (A)		1	0.9951	0.998	0.9979	0.9815	-1	1	0.961	1	1	1	1	1	1	1
9. Intercept (B)		0	-2.7	-13	14.9	-0.0395	0	0	0.07	0	0	0	0	0	0	0
10. Global Ch#		0	1	2	3	4	5	6	7	8	9	10	11	12	12	57
11. Limit: Min		3	-50	-20	-200	5	-8000		5	10	10					-5
12. Limit: Max		50	5000	4000	5000	50	500		7	90	35					50
13. Hysteresis		3	100	100	200	3	1000		0.5	5	3					3
14. Email DO#		2(<.32)	2	2	2	2	2		2	2	2					2

Figure 3. The AirDAC Config template.xls file.

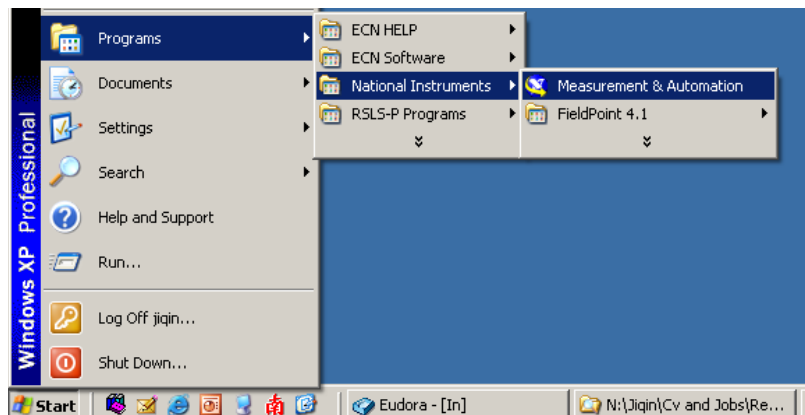



Figure 4. Run Measurement & Automation Explorer.

- 7.3.4. Locate the file. If necessary, click the  button to browse.
- 7.3.5. Click the “DAC hardware” tab (Figure 6). Select multiple FieldPoint modules of AI, TC and DO. Enter the USB device board number(s) (Refer to SOP B1 for the DAC hardware configuration and recorded board number(s)).
- 7.3.6. Click the button to save the initial configuration.
- 7.3.7. To run AirDAC, click the button in the AirDAC start screen (Figure 6). The front panel of the AirDAC will appear (Figure 7).
- 7.3.8. Activate the DAC tab (Figure 8). DAC table rows “1. Data heading” through “10.

Global ch #” are for data acquisition and calculation. Rows “11. Limit: Min” through “14. Email{ ,DO#}” are for measurement watch and device control. Rows “15. Signal” through “17. Adjusted (Y)” are for signal and calculated data monitoring.

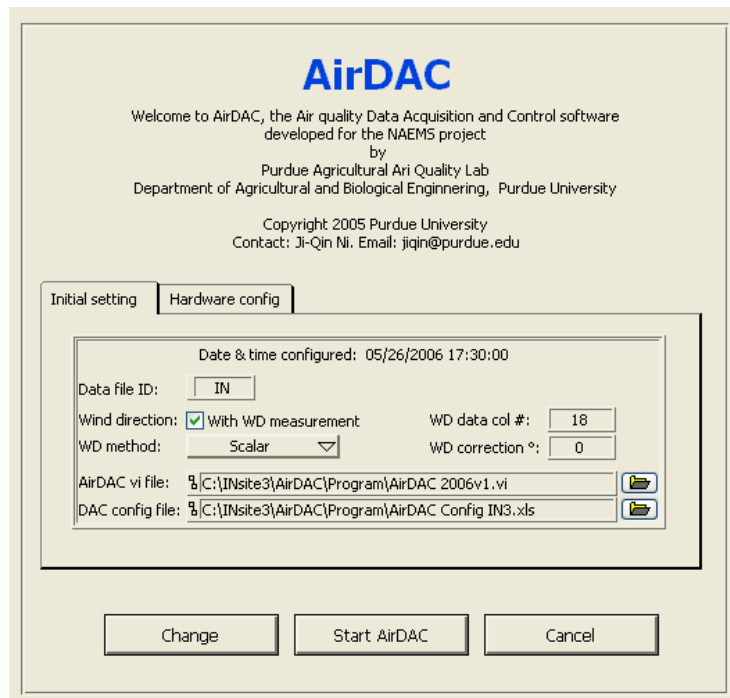


Figure 5. AirDAC start.vi with initial setting display.

- 7.3.9. Fill in the table from row “1. Data heading” through row “14. Email{ ,DO#}” according to the DAC design table (SOP B1) and the instructions in the lower part of the DAQ tab in AirDAC (Figure 8). The units in “1. Data heading” are for the “16. Converted” and “17. Adjusted” values. Leave the cells from row 2 through 14 in column 1 blank. Leave the cells from row 3 through 14 in column 2 blank. Type “0” for column 2 row 2 (Figure 8).
- 7.3.9.1. Alternatively, fill in the table in the Excel file during Step 7.1 before running AirDAC.

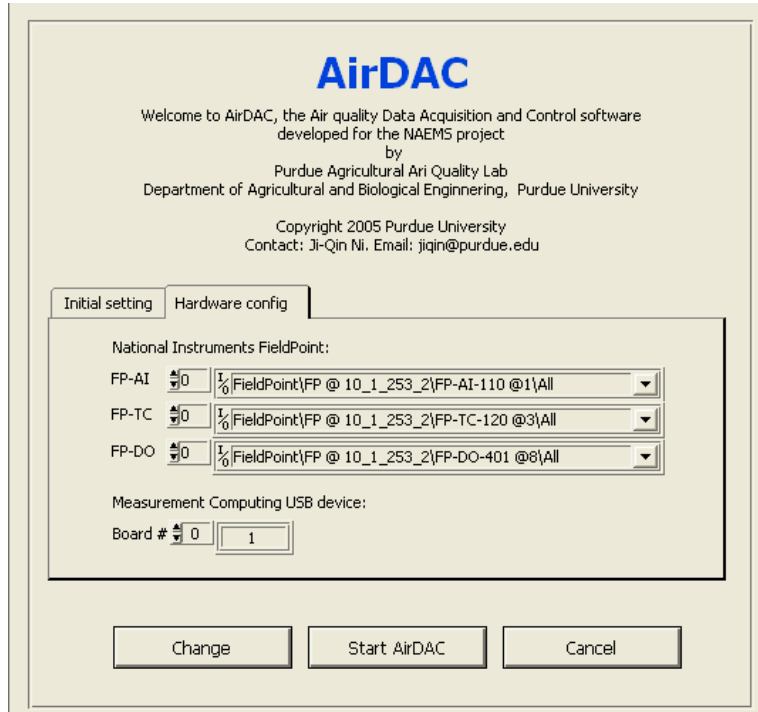


Figure 6. AirDAC start.vi with hardware configuration display.

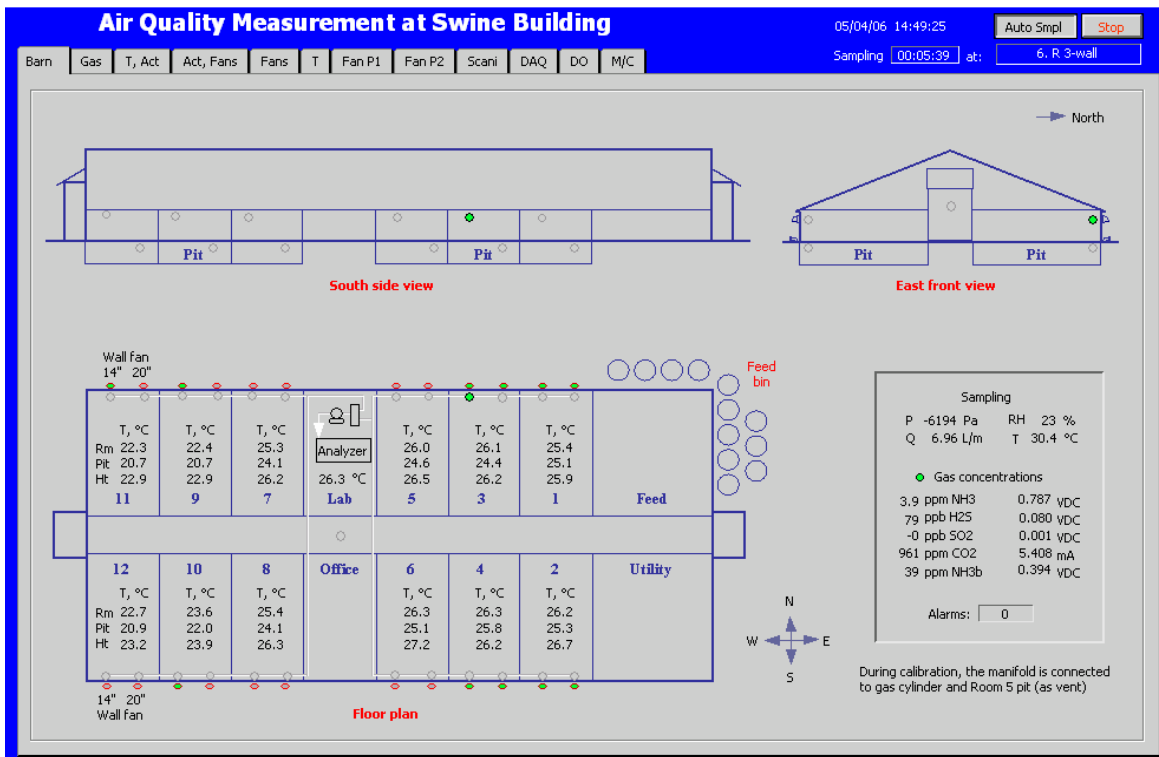


Figure 7. An example of the AirDAC front panel.

Air Quality Measurement at Swine Building

05/27/06 10:17:27 Auto Smpl

Barn Gas T, Act Act, Fans Fans T Fan P1 Fan P2 Scan DAC DO M/C Sampling 00:02:14 at: 1. R 1-pt

Monitoring/adjusting data calculation & alarm setting

	1	2	3	4	5	6	7	8	9	10	11	12
1. Data heading	Date & time	Smpl loc#	NH3, ppm	H25, ppb	SO2, ppb	CO2, ppm	NH3 M5A, ppm	Smpl P, Pa	dP smpl ex, Pa	Smpl Q, L/m	Smpl RH, %	Smpl T, °C
2. Digit of prec.		0	3	3	3	3	3	2	2		1	1
3. Signal low (SL)			0	0	0	0.004	0	0.004	0.004	0	0	0
4. Signal high (SH)			10	10	10	0.02	10	0.02	0.02	5	1	1
5. Range low (RL)			0	0	0	0	0	-34474	-37.35	0	0	-40
6. Range high (RH)			50	10000	10000	10000	1000	34474	37.35	10	100	60
7. Absolute (abs)			0	0	0	0	0	0	0	0	0	0
8. Slope (A)			1	0.9951	0.998	0.9979	0.9815	-1	1	0.961	1	1
9. Intercept (B)			0	-2.7	-1.3	14.9	-0.0995	0	0	0.07	0	0
10. Global Ch#			0	1	2	3	4	5	6	7	8	9
11. Limit: Min			3	-50	-20	-200	-5	-8000		5	10	10
12. Limit: Max			50	5000	4000	5000	50	500		7	90	35
13. Hysteresis			3	100	100	200	3	1000		0.5	5	3
14. Email{,DO#}			2	2	2	2	2	2{<,32}		2	2	2
15. Sensor sgnl (S)			0.000000	0.000000	0.000000	0.000000	0.000000	0.000000	0.000000	0.000000	0.000000	0.000000
16. Converted (X)			0.000	0.000	0.000	-2500.000	0.000	-51711.00	-56.03	0.00	0.0	-40.0
17. Adjusted (Y)			0.000	-2.700	-1.300	-2479.850	-0.100	51711.00	-56.03	0.07	0.0	-40.0

Alarms

Color

- X and Y (Y is saved in data files) are calculated with acquired sensor signal S as:
 If (abs = 0), Then X = (S - SL) / (SH - SL) * (RH - RL) + RL, Else X = abs[(S - SL) / (SH - SL) * (RH - RL) + RL]
 Y = A * X + B.
 - Absolute value (abs): 0 = false, 1 = true.
 - For Thermocouples, set SL = 0, SH = 1, RL = 0, and RH = 1.
 - For Activity sensors, which have an offset V (the sensor output = offset +/- S), set SL = offset, SH = offset + 1, RL = 0, RH = 1, A = 1, B = 0, and abs = 1.
 - Global Ch# starts from 0 and is the sequence of all local physical DAQ channels including AI, TC, and DI.
 - Min, Max, and Hysteresis are defined for Y.
 - Email{,DO#}: blank = No action, 1 = Email, 2 = Email with sampling loc#, {< = check with Min, > = check with Max, and DO#} = DO# to control (see tab "DO").
 Example: 2{<,20} = If Y < Min or Y > Max, send alarm email and indicate sampling location #; if Y < Min, turn on DO Ctrl #20.

Figure 8. An example of DAC tab in AirDAC showing some yellow-colored visual alarms when there was no DAC hardware connected.

- 7.3.10. Verify the entries and check the data displayed in rows 15 through 17 (S, X, and Y).
- 7.3.11. Click the “Apply and Save” button to save the configuration to the AirDAC Config YYSiten.xls file. When the “Apply and Save” and “Cancel changes” buttons become disabled (gray), the new configuration has been applied and saved.
- 7.3.12. Activate the DO tab (Figure 9). Click the “Change” button to activate the controls. Fill in the Name and Time, and select Control Type, Sequence, Frequency, and FP-DO channels based on the site monitoring plan, the GSS connection, and the instructions in the tab. Verify the entries and make sure that they are correct.
- 7.3.13. Click “Apply and Save” to save the configuration to the AirDAC Config YYSiten.xls file. When the “Apply and Save” and the “Cancel changes” buttons become disabled (gray), the new configuration has been applied and saved. Click the “Change” button to disable and protect the settings.
- 7.3.14. Activate the M/C tab (Figure 10). Fill in the Mail server & return address. Fill in the Email recipient’s address and define the email types that each recipient will receive.
- 7.3.15. Include odorair@ecn.purdue.edu as one of the data file recipients to deliver daily data files to PAAQL.
- 7.3.16. Verify the entries and make sure that they are correct.
- 7.3.17. Click the “Apply and Save” button to save the configuration to the AirDAC Config YYSiten.xls file. When the “Apply and Save” and the “Cancel changes” buttons become disabled (gray), the new configuration has been applied and saved.
- 7.3.18. To stop AirDAC, press the button in the upper right corner of the front panel window. In the dialog window that appears, select OK.

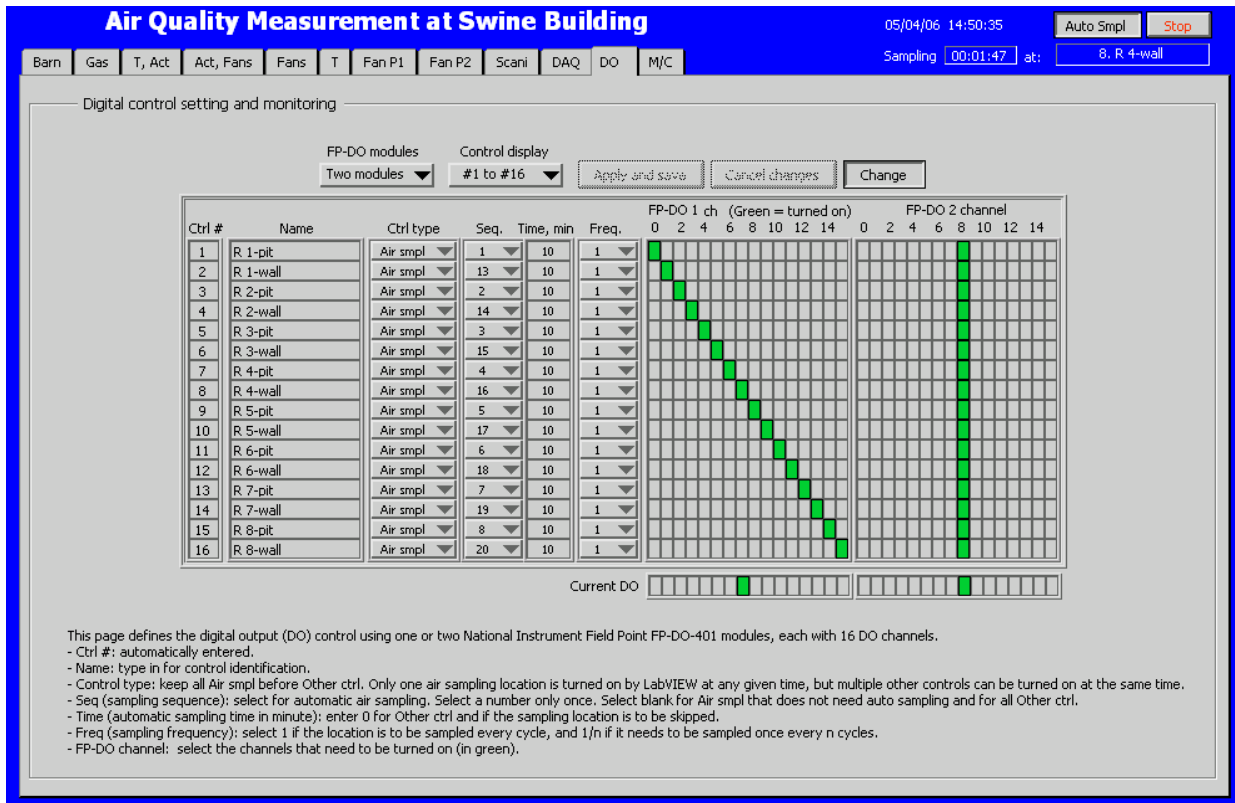


Figure 9. An example of DO tab in AirDAC.

7.4. Running AirDAC (subsequent operation): Repeat Steps 7.3.1 and 7.3.18.

7.5. AirDAC status and DAC system monitoring

7.5.1. When AirDAC is running, go to tab “M/C” (Figure 10). In “Monitoring”, verify the AirDAC loop timing. The “DAC loop t” should be 1.000 ± 0.005 s. The “Display loop t” should be 0.500 ± 0.005 s, except when AirDAC is sending email.

7.5.2. In “Monitoring” verify the AirDAC sample timing at the “sFile smpl” (which should count 15 sample numbers at 1 sample per s) and “mFile smpl” (which should count 60 sample numbers).

7.5.3. Verify the displays in the schematic representations of the barns (Figure 7).

7.5.4. Verify the other tabs for displayed measurement histories (Figure 11) to make sure that the DAC system is running properly.

7.6. Manual control of sampling locations

7.6.1. Click the button at the upper right corner of the AirDAC front panel to switch to Manual Sampling. Click the button that appears and select the location to be sampled (Figure 12).

7.6.2. To switch back to auto sampling, click the button.

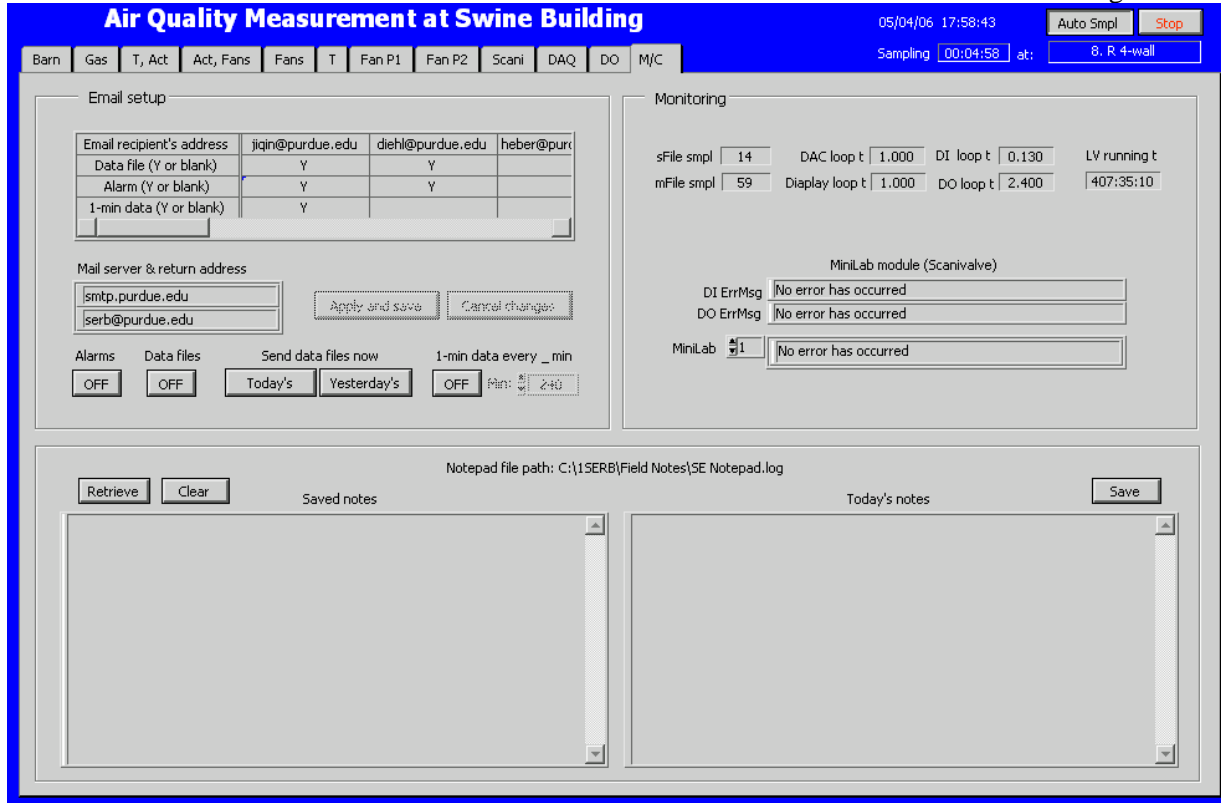


Figure 10. An example of M/C tab in AirDAC.

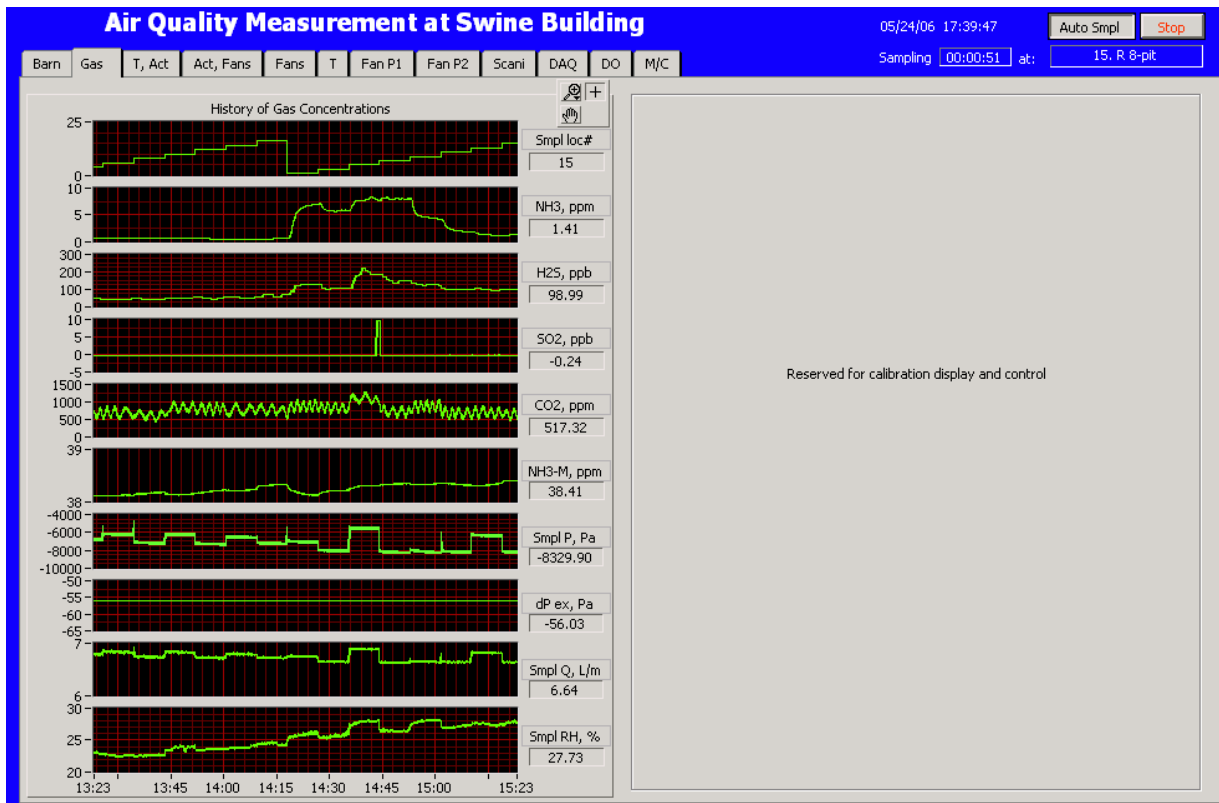


Figure 11. An example of measurement history display in AirDAC.

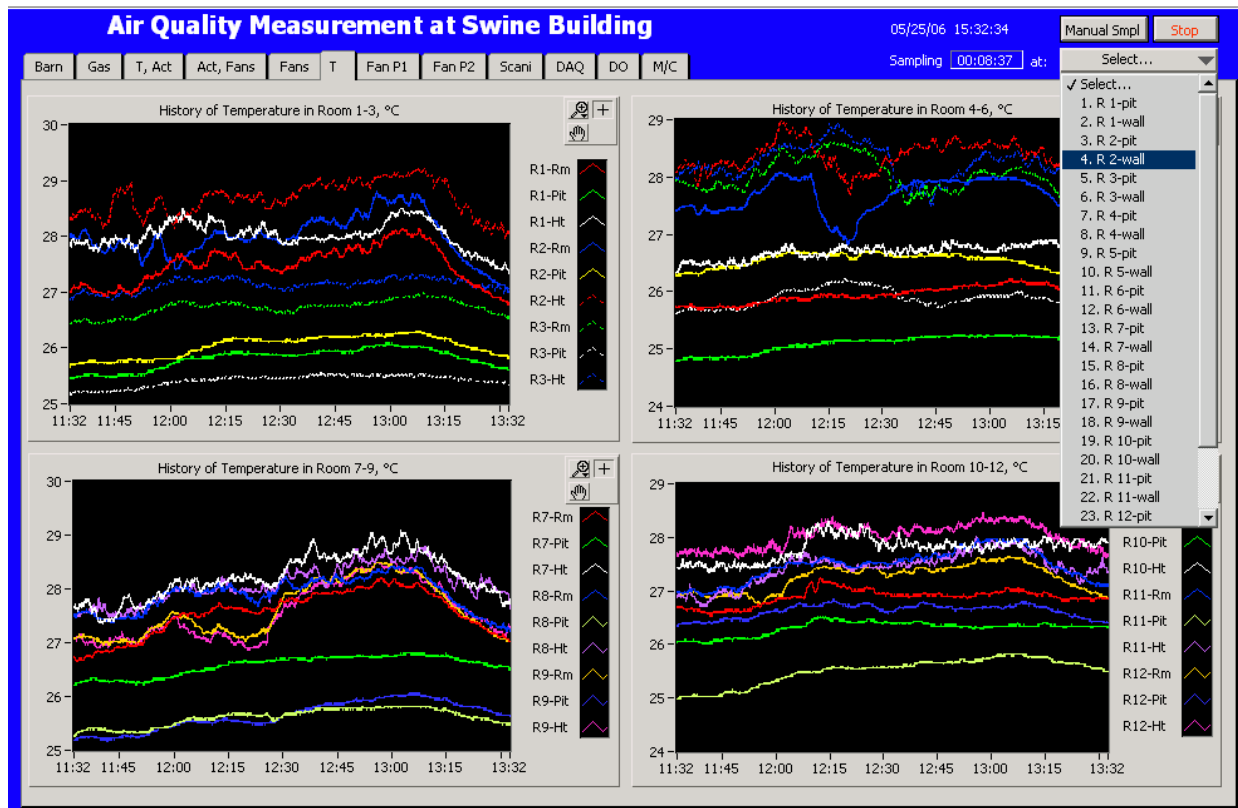


Figure 12. Manual control of sampling locations in AirDAC.

8. Data and Records Management

- 8.1. Save all AirDAC-related files in the folder C:\YYsiten\AirDAC\.
- 8.2. AirDAC initial and hardware configuration files are automatically generated and saved in C:\YYsiten\AirDAC\Program\AirDAC ini.txt. All previous configurations are also kept in the same file for record keeping and data analysis purposes.
- 8.3. AirDAC data acquisition and control configuration data are updated and saved in AirDAC Config YYsiten.xls. All previous configurations are kept in the same file. It is recommended that this file be saved in C:\YYsiten\AirDAC\Program\.
- 8.4. The daily data files will be saved in the folder C:\YYsiten\AirDAC\Data\.
- 8.5. Manage all AirDAC-related files according to SOP B5.

9. Quality Control and Quality Assurance

- 9.1. Use the built-in features in AirDAC to ensure DAC consistency and quality.
- 9.2. Keep and backup old configuration files when creating new configuration files. When editing the files, avoid altering any configuration information related to real measurement.
- 9.3. Set up alarms (rows 11 to 13 in the DAC tab, Figure 8) for real-time monitoring of all primary measurement variables, and use the alarm email feature to provide an alert for any out-of-range values for these variables. Consult the QAPP to identify the primary measurement variables.

- 9.4. Because an absence of alarm emails does not necessarily mean that the system is running normally (especially when the internet connection or the computer fails, or the AirDAC is shut down), use the “1-min data file every _min” option (Figure 10) to constantly email partial measurement data at selected time intervals. Failure to receive these emails indicates internet or measurement system trouble.
- 9.5. Maintain all records in a worksheet of the designated electronic field notes spreadsheet. Record observations and changes that can affect the measurement data.
- 9.6. Save screen shots of the AirDAC front panel displays to a PowerPoint or MS Word file when any abnormality is noticed that would not be preserved in the 1-min data files. An example of this would be a certain temperature fluctuating more than a couple of degrees C every second. The information is vital to data analysis and interpretation, but non-reproducible. To copy screen, activate the AirDAC front panel, hold the “Alt” key and press the “Print Screen” key. To paste the screen, activate the PowerPoint or MS Word file. Hold the “Ctrl” key and press “V” key.
- 9.7. Include date at the end of file name in the following format: mm-dd-yy, ex: 01-09-06. Leading zeros in day and month can be omitted, ex. 1-9-06.
- 9.8. Check the DAC system at least daily, either during a site visit, or remotely.
 - 9.8.1. Always check for displayed alarms (Figure 7 and Figure 8).
 - 9.8.2. Use the Notepad function of AirDAC (Figure 10) to record important observations and findings, and to notify other research personnel.
- 9.9. Avoid running other software that utilizes significant CPU power and memory, because sampling by AirDAC might be slowed down.
- 9.10. Seek technical support from PAAQL for problems related to AirDAC.
- 9.11. Record and report all modifications made to electronic files. The note should include the time, name of personnel, observation, changes made, and the reason.
- 9.12. Verify the conversion and adjustment of data by AirDAC before starting data collection
 - 9.12.1. Apply a known voltage or current to an analog input channel in a FieldPoint module that is configured for voltage or current signals.
 - 9.12.2. Run AirDAC.
 - 9.12.3. Configure the signal range, measurement range, slope, and intercept in the DAC tab in AirDAC. Apply and Save the configuration.
 - 9.12.4. Watch the S, X, and Y values displayed in AirDAC, and check their mathematical relationship according to the equations provided in the DAC tab. If their relationship does not agree with the equations, contact PAAQL for troubleshooting.
 - 9.12.5. After saving the data for a few min, stop AirDAC and open the data file. Find the data column that includes the test data. Compare the saved data and the Y values displayed in AirDAC. If they are different, contact PAAQL for troubleshooting.

10. References

- 10.1. SOP B1. 2006. Data Acquisition and Control Hardware. Standard Operating Procedure B1. Purdue Ag Air Quality Lab.
- 10.2. SOP B3. 2006. Air Data Pre-Processing Software. Standard Operating Procedure B3. Purdue Ag Air Quality Lab.
- 10.3. SOP B4. 2006. Calculation and Reporting of Air Emissions from Barns. Standard Operating Procedure B4. Purdue Ag Air Quality Lab.

- 10.4. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 10.5. SOP B6. 2006. Data Processing Software (CAPECAB). Standard Operating Procedure B6. Purdue Ag Air Quality Lab.
- 10.6. SOP G1. 2006. Gas Sampling System. Standard Operating Procedure G1. Purdue Ag Air Quality Lab.
- 10.7. LabVIEW manuals. Available in C:\Program Files\National Instruments\LabVIEW 7.1\manuals\
- 10.8. Webmet. 2005.http://www.webmet.com/met_monitoring/621.html (accessed 06/2005)

11. Contact Information

- 11.1. PAAQL: odor@purdue.edu, heber@purdue.edu, <http://www.AgAirQuality.com>.
- 11.2. National Instruments Corporation:
11500 N Mopac Expwy
Austin, TX 78759-3504
Technical Support: 800-531-5066.
- 11.3. Measurement Computing Corporation:
10 Commerce Way
Norton MA 02766
Phone: 508-946-5100
Fax: 508-946-9500
Email: info@mccdaq.com
- 11.4. Bugs found in the AirDAC program and suggested improvements can be directed by email (jqin@purdue.edu) or phone (765-496-1733) to Ji-Qin Ni, Purdue University.

AIR DATA PRE-PROCESSING SOFTWARE
Standard Operating Procedure (SOP) B3

AIR DATA PRE-PROCESSING SOFTWARE

Standard Operating Procedure (SOP) B3

Prepared by

Ji-Qin Ni and Juan Carlos Ramirez

Reviewed by

Teng-Teoh Lim, Albert J. Heber and Bill W. Bogan

Effective Date: November 6, 2006

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability.....	3
2. Summary of Method.....	3
3. Definitions.....	4
4. Interferences.....	4
5. Personnel Qualifications	4
6. Equipment and Supplies	5
7. Procedure.....	5
8. Records Management.....	17
9. Quality Control and Quality Assurance	18
10. References.....	18
11. Contact Information	18

1. Scope and Applicability

- 1.1. This method is used to inspect air emission data collected in livestock barns. Three levels of data review are used to ensure quality of barn air emission data.
 - 1.1.1. Real-time data monitoring is performed by AirDAC during data acquisition, and by the field engineers or personnel accessing AirDAC, either at the site or remotely. Signals acquired from all analyzers and sensors are converted to engineering units, and compared with pre-determined normal ranges every second. Alarms can be shown immediately and sent via email by AirDAC to indicate any out-of-range data that might be collected (SOP B2).
 - 1.1.2. Pre-processing of collected data into daily figures and mean value tables using Air Data Pre-Processing (ADPP) software is performed at any time after a day's data is collected. Data for related parameters are compared with each other; for instance, temperature versus barn airflow rate, and airflow versus pollutant concentrations. Hourly or daily means are compared with historical data.
 - 1.1.3. Final processing of the complete dataset is conducted using CAPECAB (SOP B6).
- 1.2. This SOP describes only the second-level of data review (1.1.2) to provide timely feedback to field engineers to prevent and correct measurement errors or other problems.
- 1.3. This SOP applies specifically to the use of ADPP to preprocess data acquired and generated by the data acquisition and control software, AirDAC (SOP B2).
- 1.4. This SOP describes the June 2006 version of ADPP. Read update notes which will be supplies by PAAQL with updated versions of ADPP for additional instructions.

2. Summary of Method

The ADPP, a macro program contained in the Air Data Pre-Process.xls file, written in Visual Basic for Applications (Microsoft Corporation), was developed by PAAQL. The ADPP uses an Excel graph template to organize raw data, assemble one or more data files in a single day, and produce daily figures as defined in the template. The ADPP also uses precision check (or zero/span check) data (SOP G9) to adjust gas concentrations or other sensor data according to the degree of instrument drift determined by these checks. In addition, the program extracts equilibrium data from location-shared analyzers and sensors (LSAS), calculates daily mean values for rapid data evaluation, and assembles multi-day mean data in a result file (Fig. 1).

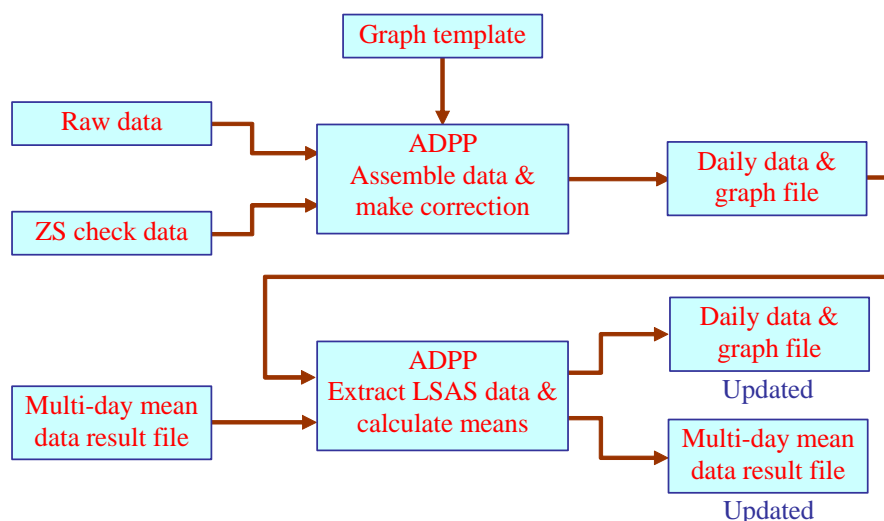


Figure 1. Data flow diagram when using ADPP to process data.

3. Definitions

- 3.1. ADPP Air Data Pre-Processing software, written in Visual Basic for Applications in MS Excel
- 3.2. AirDAC Air quality monitoring Data Acquisition and Control program, written in LabVIEW. The AirDAC program (SOP B2) is also used to display real-time data, deliver data via email, and provide information on the DAQ system's operation status to research personnel.
- 3.3. DAQ Data acquisition
- 3.4. LabVIEW Laboratory Virtual Instrumentation Engineering Workbench. A platform and development environment for a visual programming language (a dataflow language), produced by National Instruments Corporation (Austin, TX)
- 3.5. LSAS Location-shared analyzers and sensors
- 3.6. MS Microsoft Corporation
- 3.7. PAAQL Purdue Agricultural Air Quality Laboratory
- 3.8. PC Personal computer

4. Interferences

- 4.1. Interferences may occur if the computer running the ADPP is also running other software. This can result in a run-time error in the ADPP. If this occurs, it will be necessary to close other software before restarting ADPP.

5. Personnel Qualifications

- 5.1. Personnel must have good computer skills with MS Excel, including chart functions.

- 5.2. All personnel involved in data processing must read and understand this SOP, any updated information that has been supplied with newer versions of the software, and SOPs B1, B2, and B4 to know the methods of data acquisition and data processing.
- 5.3. Personnel involved in data inspection and review should have basic knowledge of AFO (animal feeding operation) emission measurement and the general ranges of measured variables, and understand the data patterns (e.g. diurnal and seasonal changes) and the relationship among the data (e.g. effect of ventilation on concentrations), so that they can correctly identify any field measurement problems.

6. Equipment and Supplies

- 6.1. PC with 1.5 GHz or higher CPU speed, 512 MB or more RAM, and 2 GB or more free hard disk space.
- 6.2. Software and files
 - 6.2.1. Windows XP and MS Excel 2003
 - 6.2.2. Air Data Pre-Process.xls file that contains the executable ADPP program
 - 6.2.3. Site-specific graph template.xls, which is created by modifying the Graph template.xls file provided by PAAQL. This will store processed data and figures, and be used by ADPP for generating uniform daily data files.
 - 6.2.4. Site-specific precision check data file, created by modifying the precision check data template.xls provided by PAAQL and containing precision check data generated during precision checks, to be used by ADPP for concentration data correction.
 - 6.2.5. Site-specific Result.xls file, which is created by modifying the Result template.xls file provided by PAAQL, to store multi-day mean data processed by ADPP, and to store data graphs.
 - 6.2.6. Raw data files in text format generated by AirDAC (SOP B2)

7. Procedure

- 7.1. Create a site-specific graph template Excel file
 - 7.1.1. Every measurement site has a different data configuration. For example, some sites may have more air-sampling locations, and others may have more measured variables. The purpose of the site-specific template is to create graphs for all the important variables at a site, and organize them for easy review and comparison (for example, keeping all the LSAS data graphs close to the location code graph), while minimizing the number of graphs.
 - 7.1.2. Create a new site-specific Excel template file, based on the existing template file Graph template.xls, which is provided by PAAQL (Fig. 2). Create a folder "ADPP" in C:\YYnX\AirDAC\ and save the "Graph template.xls" as C:\YYnX\AirDAC\ADPP\Graph YYnX.xls, where YY is the state abbreviation and n is the assigned site-specific number.

	A	B	C	D	E	F	G	H	FI	FJ	FK	FL
1	Note 1:	Measurement at RAF2. Sampling Location#: 1=B13SW,2=B14SE,3=B14Cage, 4=A										
2	Note 2:	This file is assembled with 10 LabVIEW files at 4:01:50 PM 6/14/2006. Other 5 Lat										
3	Note 3:											
4	Monday, September 15, 2003											
5	Date & Time	Smpl loc#	NH3, ppm	NO, ppm	H2S, ppb	CO2a, ppm	CO2b, ppm	Smpl Q, L/hr	Reserved	Reserved	NH3, ppm	H2S, ppb
6	0:00:00	10	27.5	0	11	1569	1505	5	4.207	1.535		
7	0:01:52	8	17.4	0	12	853	876	4.8	3.493	1.077		
8	0:02:52	8	17.2	0	14	856	876	4.8	3.493	1.084		
9	0:03:52	9	16.5	0	14	806	837	4.7	3.494	1.085		
10	0:04:52	9	13.9	0	13	789	816	4.7	3.494	1.083		
11	0:05:52	9	13.5	0	11	785	812	4.7	3.493	1.087		
12	0:06:52	9	13.7	0	10	784	813	4.7	3.493	1.092		
13	0:07:52	9	13.6	0	11	783	810	4.7	3.493	1.1		
14	0:08:52	9	13.4	0	14	787	811	4.7	3.494	1.102		
15	0:09:52	9	13.5	0	17	778	808	4.7	3.494	1.093		
16	0:10:52	9	13	0	16	779	805	4.7	3.494	1.085		
17	0:11:52	9	13.2	0	12	782	812	4.7	3.494	1.059		
1437	23:51:00	8	20.8	0	7	1239	1213	4.8	4.208	1.551		
1438	23:52:00	8	20.6	0	10	1235	1209	4.8	4.209	1.57		
1439	23:53:00	8	20.7	0	13	1231	1205	4.8	4.209	1.57		
1440	23:54:00	10	22	0	12	1480	1377	5	4.208	1.55		
1441	23:55:00	10	27.3	0	13	1573	1499	5	4.208	1.535		
1442	23:56:00	10	28.1	0	15	1601	1523	5	4.208	1.51		
1443	23:57:00	10	28	0	14	1639	1566	5	4.209	1.534		
1444	23:58:00	10	28.3	0	12	1625	1549	5	4.207	1.56		
1445	23:59:00	10	27.7	0	12	1608	1538	5	4.207	1.551		
1446												
1447												
1448												

Figure 2. Example of the “Data” worksheet in the “Graph template.xls” Excel file.

- 7.1.3. Select a range from cell B2 to cell in the last column of row 1442 of a complete raw data file. Copy this data range and paste it into the worksheet “Data” in the Graph *YYnX.xls*, starting from cell B5 (keeping the time in column A). Copy the date from cell A1 in the raw data file to cell A4 of the worksheet “Data” in the Graph *YYnX.xls*. In the first three rows in the template file, Note 1 is for sampling location codes, and will be copied from raw data files by ADPP. Note 2 will be generated automatically by ADPP. Note 3 is reserved for additional data processing information (Refer to Fig. 10 for an example).
- 7.1.4. Go to the worksheet “Graphs” of the Graph *YYnX.xls*. Make necessary modifications to the graphs, including the raw data graphs (Fig. 3) and the mean data graphs (Fig. 4), by creating new graphs, adding/deleting curves to be included in the graphs, adjusting graph format, and arranging the graphs for easy visual data inspection and comparison. Save the file.

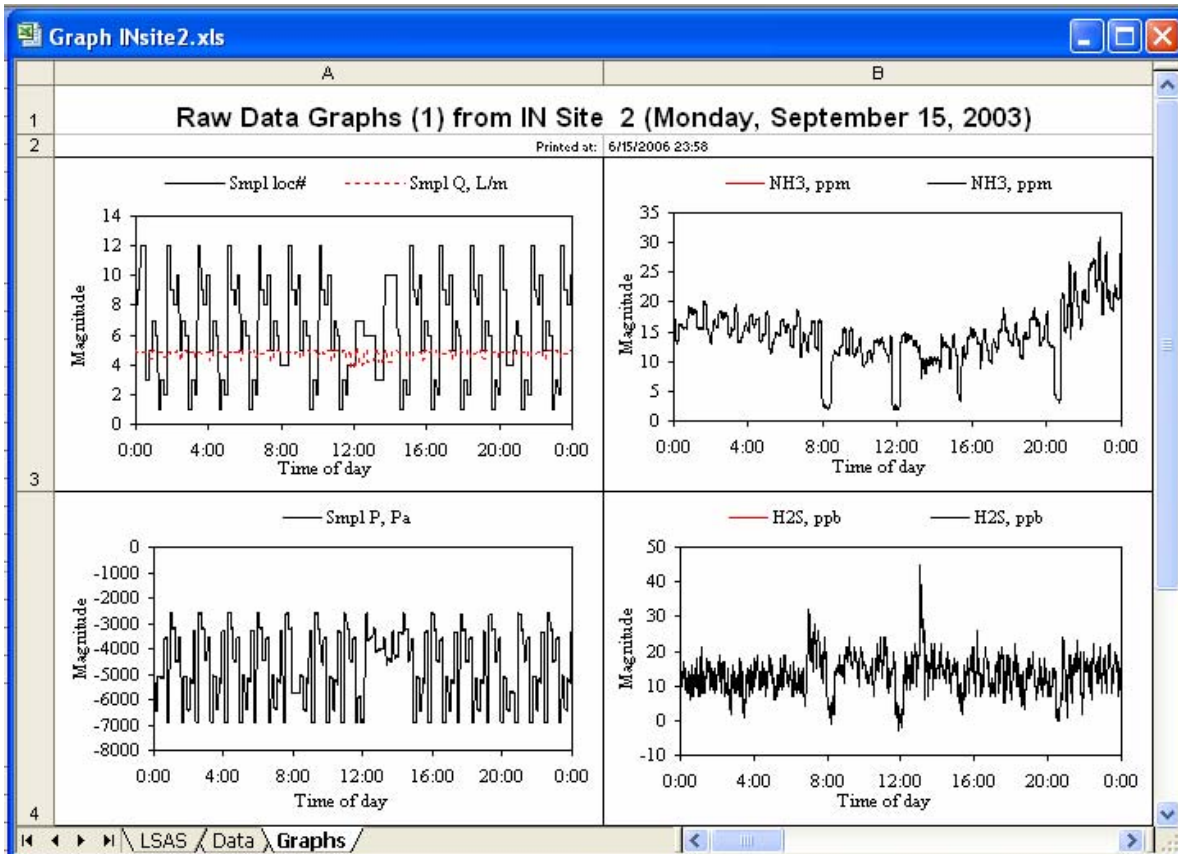


Figure 3. Example of the “Graphs” worksheet in graph template file.

- 7.1.5. The mean data graphs can also be modified after the LSAS mean data are generated in the worksheet “LSAS” by test-running the “Extract LSAS data & calculate means” feature in the ADPP (Section 7.5).
- 7.1.6. Create figures in the result file to plot mean data over days, weeks or months, so that variation can be monitored over all of these intervals.
- 7.2. Enable the “Print on Both Sides” function for raw graphs, if the printer has this option.
 - 7.2.1. It is usually desirable to print the graphs as double-sided printouts to save paper and reduce the number of hardcopies. However, this command is not available in Visual Basic for Application. Sections 7.2.2 and 7.2.3 provide an example of enabling “print on both sides” in the template file using a HP Laser Jet 8000 Series PS printer.
 - 7.2.2. Activate the Graph *YYnX.xls* file. Select File > Print... to open the Print window (Fig. 5).
 - 7.2.3. Select the printer Name. Click the “Properties...” button to activate the Properties window (Fig. 6). Select the “Layout” tab. Check “Portrait in Orientation” and “Flip on Long Edge” in “Print on Both Sides.” Click “OK” to close the Properties window. Click “Close” to close the Print window. Save the Graph *YYnX.xls* file.
- 7.3. Prepare the analyzer precision check data file.
 - 7.3.1. If gas concentration correction is to be performed by ADPP, prepare an Excel file for analyzer precision check data. This file can be created using the template file ZS

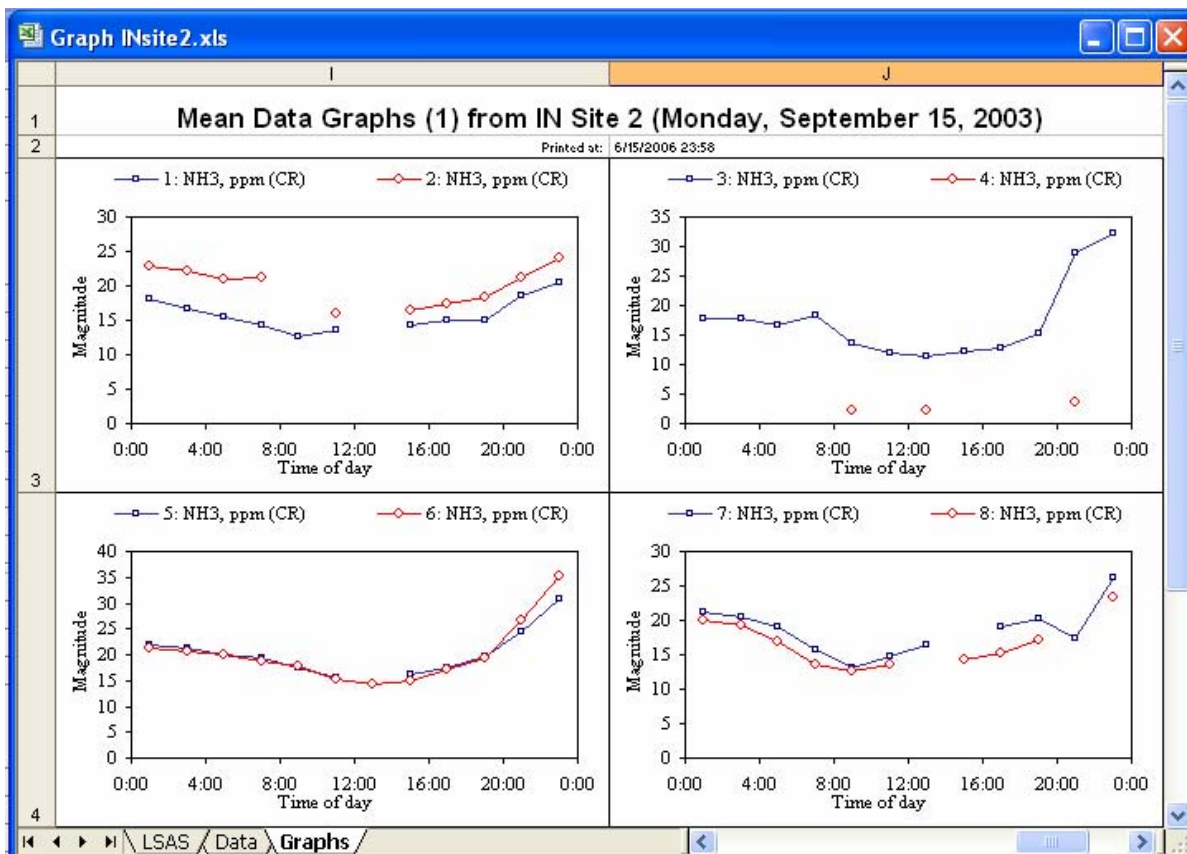
check data template.xls provided by PAAQL (Fig. 7). Follow the Notes at the top of the sheet for the format requirements of the file. Data obtained during an analyzer precision check is described in SOP G9.

7.3.2. Save the file in the folder C:\YYnX\Calibration\.

7.4. Perform "Assemble raw data and make correction."

7.4.1. Open the macro file (Air Data Pre-process.xls). If the Security Warning window (Fig. 8) appears, click "Enable Macros."

7.4.2. Click on the button in the "Start" worksheet when the Excel file opens. A user form "Select project and process" appears (Fig. 9).



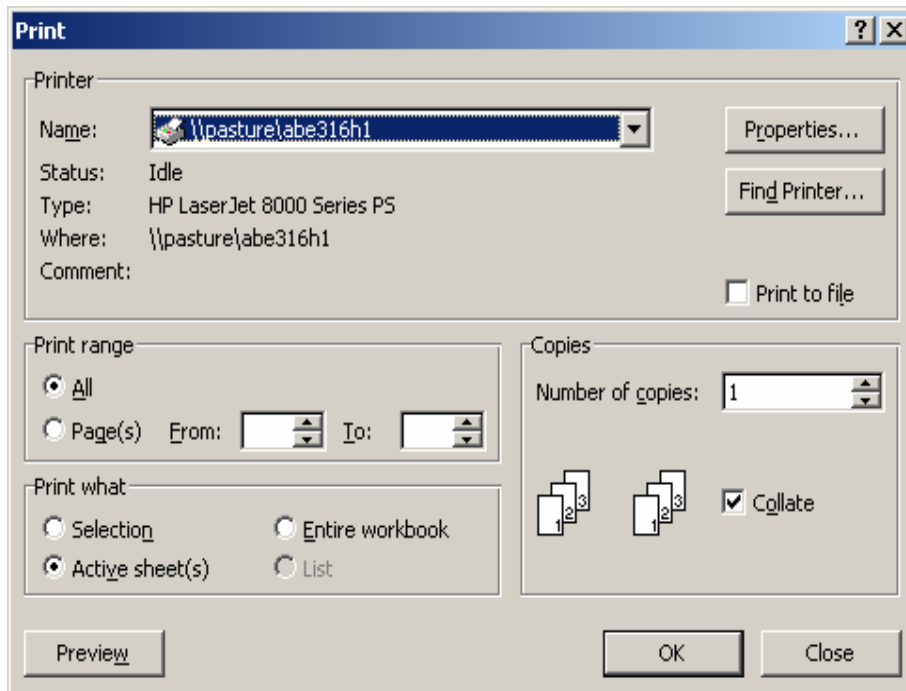


Figure 5. An example of the Print window to setup printer properties.

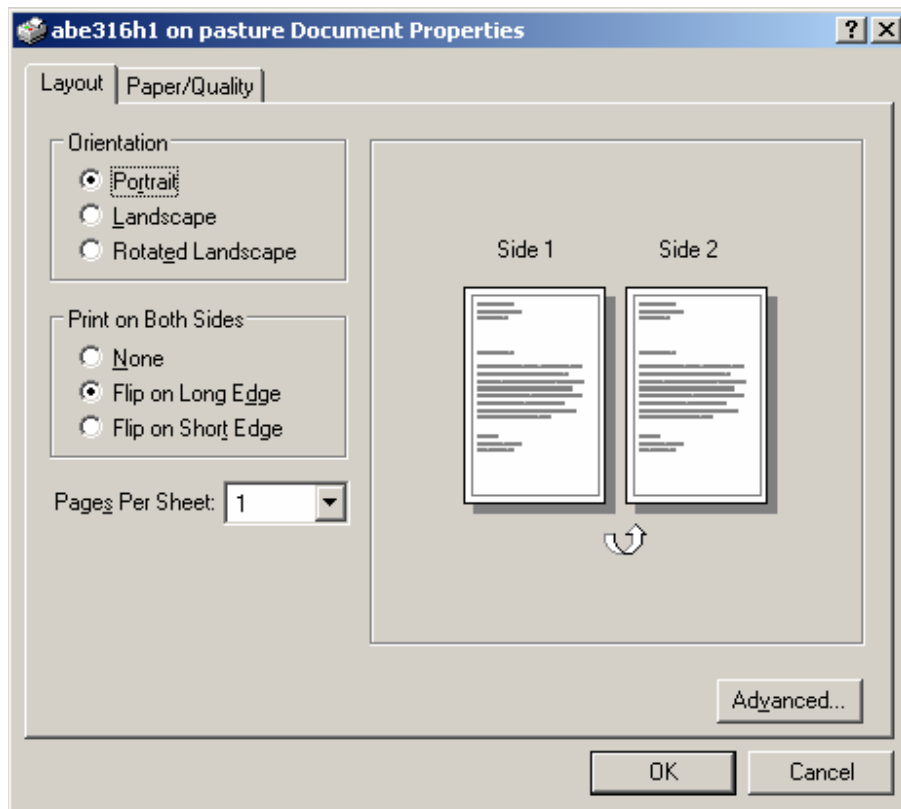


Figure 6. An example of setting up the printer properties window.

Note:
 This sheet contains precision check data that are used by Air Data Pre-Processing (ADPP) program.
 The ADPP reads the data from row 15, col 1 to row n (until there is blank cell in the date column) col 6+6n until the cell does not contain "C
 Row 13, columns 6+6n, where n is 0,1,2,..., contains data column # (C#) in the raw data files, counting "Date & time" as #1, "Smpl loc#" as #2, etc.
 For a measurement datum at time t, the Z/S check or calibration before t is the "pre check/cal," and that after t is the "post check/cal."
 Zero or span adjustment is done either at the instrument or in the AirDAC, and with Z/S gases as reference.
 If "adjusted to" was performed and has datum, "as found" before the adjustment must be recorded. If there is no adjustment, leave the cell blank.
 When the data processing program seeks the "pre-check" data, it first looks for the "adjust" data. If the "adjust" does not exist, it takes the "as found" data.
 Time, As found, and Adjust to are all the data recorded by the computer, not read in the instrument. The "Time to start" is not used in the data processing.
 When span gas cylinder is changed, the old and new cylinders' concentrations should be compared.
 Data recorded in this sheet as "As found" and "Adjust to" are the "Y" values in AirDAC (displayed in DAC page and saved in data files).

	Date and time			C3		NH3		ppm		C5		H2S		ppb		C6		CO2	
	Date	Start time	Read time	Adjust time	Zero (low), ppm	as found, ppm	adjust to, ppm	Span (high), ppm	as found, ppm	adjust to, ppm	Zero (low), ppb	as found, ppb	Zero adjust to, ppb	Span (high), ppb	as found, ppb	adjust to, ppb	Zero (low), ppm	as found, ppm	
1027	04/17/04	10:00	10:10		0	0					0	-3					0	16	
1028	04/17/04	10:10	10:20					75.3	58.5										
1029	04/17/04	10:20	10:30																
1030	04/17/04	10:30	10:40																
1031	04/17/04	10:40	10:50											903	881				
1032	04/20/04	12:20	12:30		0	0.1				0	-1						0	15	
1033	04/20/04	12:30	12:40					75.3	56.4										
1034	04/20/04	12:40	12:50																
1035	04/20/04	12:50	13:00																
1036	04/20/04	13:00	13:10											903	875				

Figure 7. Example of the analyzer precision check or zero/span check data file.

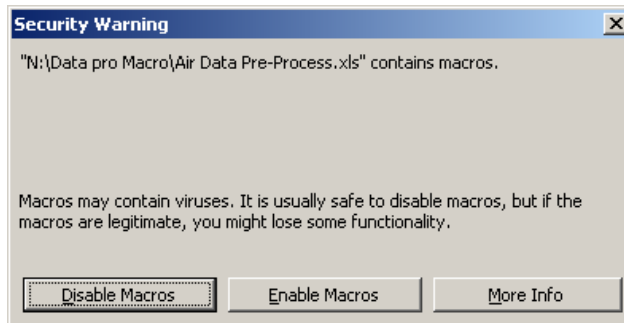


Figure 8. The Security Warning window after opening the macro file.

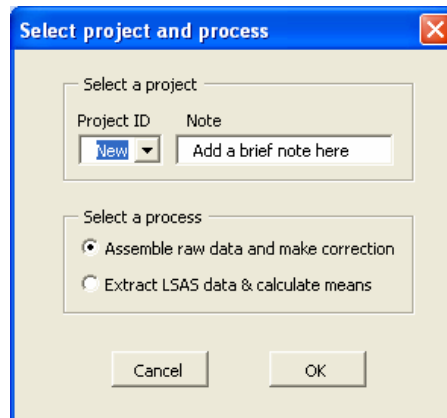


Figure 9. The “Select a project and process” user form.

- 7.4.3. At the initial use, select “New” in “Project ID” and type in a 2-letter and 1-digit site ID (for example “IN2”). Type in a brief note in the “Note” (for example “Indiana Site at RAF2”) in the “Select a project.” Select “Assemble raw data and make correction” in the “Select a process” and click OK. A new user form “Assemble Raw Files, Correct Data, and Print Graphs” appears (Fig. 10). In subsequent uses, the project ID and note entered in this step will appear as default values.

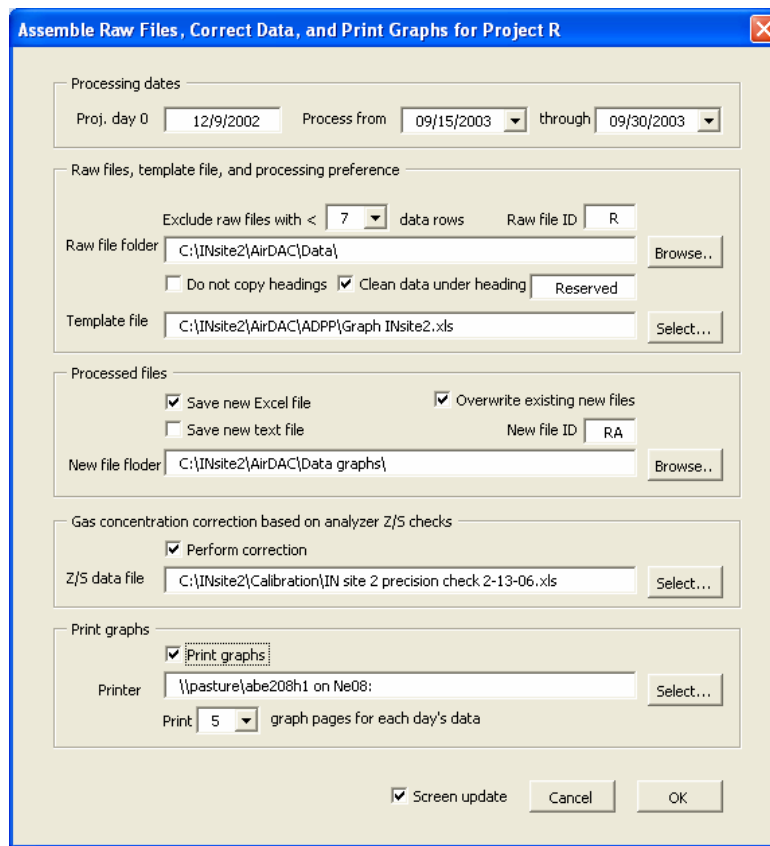


Figure 10. The “Assemble Raw Files, Correct Data, and Print Graphs” user form.

- 7.4.4. Configure processing settings in the “Assemble Raw Files, Correct Data, and Print Graphs” form.
- 7.4.4.1. Processing dates
 - 7.4.4.1.1. Type in the project’s starting day (day 0) using format “m/d/yyyy.”
 - 7.4.4.1.2. Select the dates to process in “Process from” and “through” list boxes.
 - 7.4.4.2. Raw files, template files, and processing preference
 - 7.4.4.2.1. Select the minimum number of data rows required, in order to exclude from processing any raw data files that contain too few data.
 - 7.4.4.2.2. Type a data file ID in “Raw file ID” (for example, “RA” for raw data files RAYyyymmddx.txt, where yyyymmdd are year, month and date, and x is the file serial letter of the day).
 - 7.4.4.2.3. Select the raw data file folder by clicking the “Browse...” button.
 - 7.4.4.2.4. Check “Do not copy headings” to keep the data headings in the template file. Otherwise, the headings of the first file of the day will be used.
 - 7.4.4.2.5. Check “Clean data under heading”, and type in the heading to remove useless data and reduce processed data file size.
 - 7.4.4.2.6. Find the template file by clicking the “Select...” button.
 - 7.4.4.3. Processed files
 - 7.4.4.3.1. Check “Save new Excel file” and “Save new text file” if these files are to be saved. The new Excel file will contain daily graphs and processed results. The new text file will only contain 1-min data from the “Data” worksheet.
 - 7.4.4.3.2. Enter the “New file ID”, which should be a 2-letter ID if the new text files are to be further processed by the CAPECAB data processing software (SOP B6).
 - 7.4.4.3.3. Check “Overwrite existing new files” if desired.
 - 7.4.4.3.4. Select the folder to save the new files by clicking “Browse...”
 - 7.4.4.4. Gas concentration correction based on analyzer precision checks
 - 7.4.4.4.1. Check “Perform correction” if gas concentrations are to be corrected.
 - 7.4.4.4.2. Select the precision data file by clicking “Select...” Corrected gas concentrations will be saved in the original data column, and the raw data will be moved to the right side of the sheet in the new Excel graph data file. Comments will be added in the headings of the relevant data columns (Fig. 11) and a sheet “Notes” will be added during processing.
 - 7.4.4.4.3. Gas concentration data correction requires two sets of precision check data, as only measurements made between two checks can be adjusted. Since gas analyzer precision checks are usually not conducted daily, gas concentration data cannot be corrected until the post-measurement precision check is conducted. If necessary, process the data twice using the ADPP. The first of these provides a quick data review/feedback; the second, with two complete precision check datasets, provides actual data assessment.

Date & Time	Smpl loc#	NH3, ppm (CR)	CO2b, ppm (CR)	Smpl Q, L/m Sm
0:00:00	1	15.882	571	4.9
0:01:00	10	32.232	985	5
0:02:00	10	32.815	1719.843	5
0:03:00	10	32.815	15.46	5
0:04:00	5	31.764	14.379	4.6
0:05:00	5	30.129	1688.029	4.4
0:06:00	5	31.413	1638.847	4.4
0:07:00	5	30.128	17.626	4.4
0:08:00	5	30.478	19.79	4.4
0:09:00	5	30.361	18.71	4.4
0:10:00	5	30.828	18.71	4.4
0:11:00	5	30.127	17.63	4.4
0:12:00	5	30.944	18.712	4.4
0:13:00	5	31.178	23.039	4.4
0:14:00	7	28.959	21.959	5
23:22:00	10	16.662	11.246	5
23:23:00	10	16.777	10.166	5
23:24:00	5	17.353	12.329	4.6
23:25:00	5	21.269	14.493	4.4
23:26:00	5	21.73	13.412	4.4
23:27:00	5	21.729	11.25	4.4
23:28:00	5	21.499	12.333	4.4
23:29:00	5	22.075	12.334	4.4
23:30:00	5	22.305	11.253	4.4

Figure 11. Original and adjusted gas concentrations in the Excel graph file.

7.4.4.5. Printing graphs:

7.4.4.5.1. Check “Print graphs” if the figures in the “Graphs” sheet are to be printed.

7.4.4.5.2. Select the printer by clicking the “Select...” button.

7.4.4.5.3. Select the number of pages to be printed, based on the template file configuration.

7.4.5. Check “Screen update” if the processing is to be monitored. More time will be required to finish the data pre-processing if this option is selected. When all settings are correctly made, click “OK” to start processing.

7.5. Perform “Extract LSAS data and calculate means.”

7.5.1. After the “Assemble raw data and make correction” process is completed, ADPP can be used to extract the equilibrated data from LSAS (mainly gas analyzers and sensors in the GSS) and to calculate mean data.

7.5.2. Repeat the steps in Sections 7.4.1 (if the macro file is to be opened) and 7.4.2. The “Select project and process” user form appears (Fig. 12).

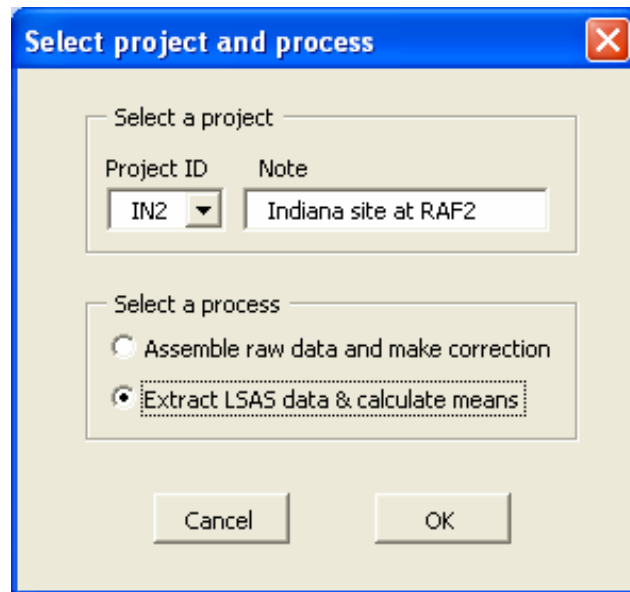


Figure 12. Select project and process for extract LSAS and get mean data.

- 7.5.3. Select the Project ID, check “Extract LSAS and calculate means,” and click OK. A user form “Extract Data from LSAS and Calculate Means” appears (Fig. 13).
- 7.5.4. Configure processing settings in the “Extract Data from LSAS and Calculate Means” form.
 - 7.5.4.1. Processing dates
 - 7.5.4.1.1. Type in the project starting day (day 0) using the “m/d/yyyy” format. This date will also be used by ADPP to determine the starting row of the mean data in the result file (Section 7.5.4.4).
 - 7.5.4.1.2. Select the range of dates to process in “Process from” and “through” list boxes.
 - 7.5.4.2. Select folders, files and processing preference
 - 7.5.4.2.1. Select the file folder where the processed Excel files were saved during the “Assemble raw data and make corrections” processing by clicking “Browse...” Enter the “File ID”.
 - 7.5.4.2.2. Select between “Save update in original Excel files” and “Save update in new Excel file.” If the latter is selected, enter the “New file ID” and select the new file folder.
 - 7.5.4.2.3. Check “Overwrite existing files” and “Save update in new text file” as desired.
 - 7.5.4.3. Extract valid data from LSAS
 - 7.5.4.3.1. Check “Perform extraction” and enter the “Data Column #”, which is the column # in the raw data file (column # 1 is date & time, column # 2 is sampling location code, and so on), and “Valid t, min”, which is the last n minutes in the same sampling location (for example, in the 10-min sampling, entering “3” in the “Valid t, min” will extract the 8th – 10th min data). Refer to SOP B4 for amount of data (i.e. the number of minutes) which are taken as valid for different gases

Extract Valid Data from LSAS and Calculate Means for Project R

Processing dates
 Project day 0: 9/15/2003 Process from: 09/15/2003 through: 09/30/2003

Select folders, files and processing preference
 Save update in original Excel files File ID: RA
 Excel file folder: C:\INsite2\AirDAC\Data graphs\ Browse...
 Save update in new Excel file Overwrite existing files
 Save update in new text file New file ID: RB
 New file folder: C:\INsite2\AirDAC\Data graphs\ Browse...

Extract valid data from location-shared analyzers/sensors
 Perform extraction

Data column #	3	5	6	7	42	8	9			
Valid t, min	3	4	5	5	3	3	3			

 Total smpl loc: 17 Clean data during calibration when Loc # is: 16

Update results
 Update result file
 Result file: C:\INsite2\AirDAC\Data results\IN site 2 result 6-15-06b.xls Select...
 Update 1-h mean Update 2-h mean Update 3-h mean
 Update 4-h mean Update 6-h mean Update daily mean

Print graphs
 Print graphs
 Printer: \\pasture\abe208h1 on Ne08: Select...
 Print: 8 graph pages in "Graphs" sheet for each day's data

Screen update Cancel OK

Figure 13. User form to set the LSAS data extraction and mean data calculation.

- 7.5.4.3.2. Enter the number of sampling locations in the “Total smpl loc”; this is used by the software to determine the size of the mean data table.
- 7.5.4.3.3. Enter the location number of analyzer calibration if the data during calibration should be cleared.
- 7.5.4.4. Update results
 - 7.5.4.4.1. Check “Update result file” if the mean data in daily files are to be copied into a separate result file by the ADPP.

- 7.5.4.4.2. Check the different mean data as desired. New worksheets will be added into the result file by the ADPP based on these selections.
- 7.5.4.5. Printing graphs
 - 7.5.4.5.1. Check “Print graphs” if the figures in the “Graphs” sheet are to be printed.
 - 7.5.4.5.2. Select the printer by clicking the “Select...” button, and select the number of pages to be printed based on the template file configuration.
- 7.5.5. Check the “Screen update” if the process is to be monitored. Click the OK button to start processing when all settings are correctly made.
- 7.5.6. An example of extracted data from LSAS in daily Excel files is provided in Fig. 14, and an example of a processed result file containing multi-day data is illustrated in Fig. 15.
- 7.6. Troubleshooting
 - 7.6.1. If the program displays an error message, first check the raw data files. The format of the raw data files should be the same as the one generated by AirDAC (Fig. 16), in which cell 1A contains the date, cell 1D stores the sampling location codes, row 2 has data headings, and data are saved from row 3 on. Delete the empty raw data files or correct the file format, and try running the program again.
 - 7.6.2. Excel spreadsheets allow a maximum of 256 columns. A run-time error will occur if the number of data columns exceeds 256. Reduce the number of columns and try running the program again.
 - 7.6.3. For errors that cannot be solved, contact jiqin@purdue.edu for troubleshooting.

RA20030916.xls												
1	Note: This sheet contains extracted data from location-shared analyzers and sensors processed at 2:09:18 PM E											
2												
3												
4	Tuesday, September 16, 2003											
5	Date & Time	Smpl loc#	NH3, ppm (CR)	H2S, ppb (CR)	CO2a, ppm (CR)	CO2b, ppm (CR)	MSA_NH3 (CR)	Smpl G, L/h	Smpl P, Pa			
6	0:00:00	1		11.131	782.571	795.185						
7	0:01:00	10	32.232	15.458	1686.985	1606.586	0.259	5	-3349			
8	0:02:00	10	32.815	17.622	1715.843	1635.907	0.259	5	-3358			
1493	4-hour mean	Time	1: NH3, ppm (CR)	2: NH3, ppm (CR)	3: NH3, ppm (CR)	4: NH3, ppm (CR)	5: NH3, ppm (CR)	6: NH3, ppm (CR)	7: NH3, ppm (CR)	10: Smpl P, Pa	11: Smpl P, Pa	12: Smpl P, Pa
1494	9/16/2003 2:00	2:00	21.12	27.59	35.49		31.72	39.89	26.9	-3353.56		-5087.5
1495	9/16/2003 6:00	6:00	16.19	24.12	28.37		25.23	28.1	19.32	-3334.5		-5079
1496	9/16/2003 10:00	10:00	14.15	17.94	14.93	1.42	20.41	20.33	15.8	-3340.78		-5173
1497	9/16/2003 14:00	14:00	15.2	18.91	14.8		20.47	18.01	19.21	-3406.33		-5204
1498	9/16/2003 18:00	18:00	16.31	18.73	15.44		21.88	19.55	22.44	-3389.67		-5205.67
1499	9/16/2003 22:00	22:00	16.85	18.44	16.65	0.53	21.63	20.23	23.97	-3356.83		-5165.33
1500	6-hour mean	Time	1: NH3, ppm (CR)	2: NH3, ppm (CR)	3: NH3, ppm (CR)	4: NH3, ppm (CR)	5: NH3, ppm (CR)	6: NH3, ppm (CR)	7: NH3, ppm (CR)	10: Smpl P, Pa	11: Smpl P, Pa	12: Smpl P, Pa
1501	9/16/2003 3:00	3:00	19.41	28.09	33.67		30.59	37.88	25.61	-3350.75		-5084
1502	9/16/2003 9:00	9:00	13.66	20.41	18.17	1.42	21.13	21.68	16.16	-3337.25		-5126.5
1503	9/16/2003 15:00	15:00	15.53	19	15.09		21.02	18.43	19.96	-3409.33		-5206.17
1504	9/16/2003 21:00	21:00	16.6	18.29	16.09	0.53	21.63	20.02	23.64	-3359.22		-5177
1505	Daily mean	Time	1: NH3, ppm (CR)	2: NH3, ppm (CR)	3: NH3, ppm (CR)	4: NH3, ppm (CR)	5: NH3, ppm (CR)	6: NH3, ppm (CR)	7: NH3, ppm (CR)	10: Smpl P, Pa	11: Smpl P, Pa	12: Smpl P, Pa
1506	9/16/2003 12:00		16.68	21.26	21.51	0.98	23.76	24.69	21.69	-3361.26		-5150.98
1507	Sampling t, min	Loc# 1	Loc# 2	Loc# 3	Loc# 4	Loc# 5	Loc# 6	Loc# 7	Loc# 8			
1508	9/16/2003	137	130	130	50	150	150	150	130			

Figure 14. Example of the extracted LSAS data and the mean data separated by sampling locations. The “Time” column is for graph plotting.

5	Date and time	Time	1: NH3, ppm (CR)	2: NH3, ppm (CR)	3: NH3, ppm (CR)	4: NH3, ppm (CR)	5: NH3, ppm (CR)	10: Smpl P, Pa	11: Smpl P, Pa	12: Smpl P, Pa
6	9/15/2003 3:00	3:00	16.72	21.94	17.6		20.8	-3296.6		-5106.42
7	9/15/2003 9:00	9:00	13.49	18.54	14.71	2.34	17.47	-3306.11		-5102.89
8	9/15/2003 15:00	15:00	14.64	16.91	12.05	2.12	16.9	-3358		-5147.17
9	9/15/2003 21:00	21:00	17.31	21.14	22.99	3.58	25.05	-3347		-5127.25
10	9/16/2003 3:00	3:00	19.41	28.09	33.67		30.59	-3350.75		-5084
11	9/16/2003 9:00	9:00	13.66	20.41	18.17	1.42	21.13	-3337.25		-5126.5
12	9/16/2003 15:00	15:00	15.53	19	15.09		21.02	-3409.33		-5206.17
13	9/16/2003 21:00	21:00	16.6	18.29	16.09	0.53	21.63	-3359.22		-5177
14	9/17/2003 3:00	3:00	14.22	20.22	18.11		22.36	-3341.56		-5112.92
15	9/17/2003 9:00	9:00	12.67	15.38	15	1.41	18.15	-3376.58		-5161.44
63	9/29/2003 9:00	9:00	25.28	31.28	26.84	1.7	31.12	-4130.83	-3189.33	-5296.86
64	9/29/2003 15:00	15:00	14.89	19.69	20.15		22.91	-4145.11		-5323.78
65	9/29/2003 21:00	21:00	27.12	25.9	27.84		28.92	-4067.67		-5297.22
66	9/30/2003 3:00	3:00	34.51	35.21	33.79	2.02	35.29	-4160.22		-5262.5
67	9/30/2003 9:00	9:00	22.8	28.85	26.23		31.19	-4129.67		-5308.22
68	9/30/2003 15:00	15:00	17.15	22.34	21.08	1.76	26.88	-4065		-5328.17
69	9/30/2003 21:00	21:00	28.19	23.12	24.04		27.46	-4069		-5316.08

Figure 15. Example of a result file in which mean values were calculated for different sampling locations. The “Time” column is for sorting data to evaluate diurnal patterns.

2	Date & time	Smpl loc#	dP loc#	NH3, ppm	NO, ppm	H2S, ppb	SO2, ppb	CO2, ppm	Smpl P, P df
3	5/9/2005 0:00	16	0	1.7	25.8	9	-1250	0	51710.2
4	5/9/2005 0:01	16	0	1.7	25.9	9	-1250	0	51710.2
5	5/9/2005 0:02	16	0	1.7	25.9	9	-1250	0	51710.2
6	5/9/2005 0:03	16	0	1.7	25.9	9	-1250	0	51710.2
7	5/9/2005 0:04	17	0	1.7	25.9	9	-1250	0	51710.2
8	5/9/2005 0:05	17	0	1.7	26	9	-1250	0	51710.2
9	5/9/2005 0:06	17	0	1.7	26.1	9	-1250	0	51710.2
10	5/9/2005 0:07	17	0	1.7	26.3	9	-1250	0	51710.2
11	5/9/2005 0:08	17	0	1.7	26.3	9	-1250	0	51710.2
12	5/9/2005 0:09	17	0	1.7	26.3	9	-1250	0	51710.2
13	5/9/2005 0:10	17	0	1.7	26.3	9	-1250	0	51710.2
14	5/9/2005 0:11	17	0	1.7	26.3	9	-1250	0	51710.2
15	5/9/2005 0:12	17	0	1.7	26.4	9	-1250	0	51710.2
16	5/9/2005 0:13	17	0	1.7	26.4	9	-1250	0	51710.2

Figure 16. Format of the raw data file.

8. Records Management

- 8.1. Manage data files according to SOP B5.
- 8.2. ADPP automatically records the date and time of processing and the number of raw data files used in the daily Excel file that it generates.
- 8.3. ADPP automatically records the name of the precision check file, the name of the worksheet where precision data are used for concentration correction, and the date and time the correction was made in the “Notes” worksheet in the daily Excel file.
- 8.4. Record other information including date, analyst name, site name, and narrative in the data processing notebook or computer file.

9. Quality Control and Quality Assurance

- 9.1. Verify the results of the data processing to make sure that all results are correct at the initial use of the software. This can be done by processing a few days worth of data, and compare the results (adjusted vs. original gas concentrations, extracted vs. original data for LSAS, and minute vs. averaged data) for any inconsistencies.
- 9.2. Be sure to modify the graph template file, result file, and ADPP configuration to meet the new data processing requirement and avoid incorrect presentation of the data if any change is made to the measurement configuration and data file format (e.g. adding a new sensor or changing DAQ channel(s)).
- 9.3. Provide timely feedback to the personnel maintaining the measurement site whenever an error is found and/or corrective action is needed.

10. References

- 10.1. USEPA. 2001. Guidance for Preparing Standard Operating Procedures (SOPs) EPA QA/G-6, EPA, EPA/240/B-01/004, Appendix E. Available at <http://www.epa.gov/QUALITY/qs-docs/g6-final.pdf>. Accessed on May 2005.
- 10.2. SOP B1. 2006. Data Acquisition and Control Hardware. Standard Operating Procedure B1. Purdue Ag Air Quality Lab.
- 10.3. SOP B2. 2006. Data Acquisition and Control Software (AirDAC). Standard Operating Procedure B2. Purdue Ag Air Quality Lab.
- 10.4. SOP B4. 2006. Calculation and Reporting of Air Emissions from Barns. Standard Operating Procedure B4. Purdue Ag Air Quality Lab.
- 10.5. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 10.6. SOP B6. 2006. Data Processing Software (CAPECAB). Standard Operating Procedure B6. Purdue Ag Air Quality Lab.
- 10.7. SOP G9. 2006. Precision Checks of Gas Analyzers. Standard Operating Procedure G9. Purdue Ag Air Quality Lab.

11. Contact Information

- 11.1. PAAQL: odor@purdue.edu, heber@purdue.edu, <http://www.AgAirQuality.com>.
- 11.2. For troubleshooting of ADPP: jiqin@purdue.edu

CALCULATION AND REPORTING OF AIR EMISSIONS FROM BARNES

Standard Operating Procedure (SOP) B4

CALCULATION AND REPORTING OF AIR EMISSIONS FROM BARNs
Standard Operating Procedure (SOP) B4

Prepared by

Teng-Teoh Lim and Bill W. Bogan

Reviewed by

Ji-Qin Ni and Albert J. Heber

Effective Date: November 6, 2006

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions of Terms and Acronyms	3
4. Interferences.....	4
5. Personnel Qualifications.....	5
6. Procedures	5
7. Data and Records Management	11
8. Quality Control and Quality Assurance	11
9. References.....	12
Appendix A. Formats for Reporting Daily Mean Data at Two Barns.....	13
Appendix B. Derivation of Gas Emission Equations	14
Appendix C. Correcting PM₁₀ Concentrations from the TEOM	16
Appendix D. Humidity Ratio Calculation	19

1. Scope and Applicability

- 1.1. This standard operating procedure (SOP) describes the methods for calculating and reporting gas and particulate matter (PM) concentrations and emission rates from livestock barns.
- 1.2. This method applies to continuous measurements of gas and PM emission data from livestock barns.
- 1.3. Several emission equations, along with their derivations, are included. Some discussion of CAPECAB functions that are used to perform the calculations is also included, although this is discussed in much more detail in SOP B6.
- 1.4. The equations and report formats in this SOP are used in the data processing software CAPECAB (SOP B6). They can also be used for manual calculations and report generation, or by or other computer software (e.g. MS Excel).

2. Summary of Method

The emission rate for a barn or similar structure is calculated by multiplying ventilation rate (the volume of air exiting the barn per unit time) by the contaminant concentration (specifically, the concentration difference between exhaust and inlet air). In the emission rate calculations for gases, volumetric gas concentrations (ppmv or ppbv), reported by most of the gas analyzers, are converted to mass concentrations (mg/m^3 or $\mu\text{g}/\text{m}^3$), which are then multiplied by an exhaust airflow rate (m^3/time) that is based on the same air volume density. For TSP and $\text{PM}_{2.5}$ emission rates, since the concentration is given in the form of a mass concentration based on the as-found sampling flow rate, the calculation of emission rate is straightforward. However, the PM_{10} concentration, as measured by the TEOM, is reported based on 25°C, and 1 atm of sampling flow rate; thus, this must be converted to standard temperature and pressure conditions (20°C, 1 atm) in the emission rate calculation. The ventilation rate for PM_{10} emission rate calculation is also converted to dry STP conditions.

3. Definitions of Terms and Acronyms

- | | | |
|------|--------------------|--|
| 3.1. | AirDAC | Air Data Acquisition and Control program |
| 3.2. | ASABE | American Society of Agricultural and Biological Engineers |
| 3.3. | atm | atmosphere, 746 mBar or 101,325 Pa |
| 3.4. | Average daily mean | Average of valid daily means over a certain period of time, such as a week, month, season or year |
| 3.5. | Complete Data Day | A day in which the collected and valid data cover >70% of the time (>16.8 h) |
| 3.6. | Daily mean | Average of all valid and interpolated data records during a Complete Data Day |
| 3.7. | Data record | A single data point recorded in data files by AirDAC. A data record consists of an average of 1-s readings collected over the data recording interval. AirDAC takes a reading for each measurement every s, and writes a data record 60 s, after |

	averaging 60 readings. AirDAC also writes 15-s data records, based on the averages of 15 readings.
3.8. GSS	Gas sampling system
3.9. Overall test mean	Average of all valid data over the entire test
3.10. PAAQL	Purdue Agricultural Air Quality Laboratory
3.11. Period mean	Average of valid data records during the sampling period
3.12. PM	Particulate matter
3.13. PM ₁₀	PM with a diameter $\leq 10 \mu\text{m}$
3.14. QAPP	Quality Assurance Project Plan
3.15. Reading	One data point acquired by the data acquisition or data logging program
3.16. RH	Relative humidity
3.17. Sampling cycle	The amount of time that the GSS takes to scan through all sampling locations, and return back to the starting location. For example, in a project with 12 locations, which are sampled for 10 min each, the sampling cycle is 2 h (120 min).
3.18. SLG	Sampling location group
3.19. SOP	Standard Operating Procedure
3.20. STP	Standard Temperature and Pressure. For this SOP, STP is defined as 20°C and 1 atm. “Moist STP” refers to moist air at STP conditions, while “dry STP” refers to dry air at STP conditions.
3.21. TEOM	Tapered Element Oscillating Microbalance
3.22. TM	Total mass

4. Interferences

- 4.1. Gas concentrations in volumetric units (e.g. ppmv and ppbv) and emission rates in mass/time units (e.g. g/s) are independent of air density (as influenced by temperature, pressure, humidity and barometric pressure). However, concentrations in mass units (mg/m^3 or $\mu\text{g}/\text{m}^3$) are dependent on density, and whether the volume unit refers to an air–water vapor mixture (moist air) or dry air.
 - 4.1.1. When reporting airflow rate and mass concentrations, base the air volume on dry air (0.0% RH) at STP (20°C and 1 atm).
 - 4.1.2. Overall (including both seasonal and geographical variations) outdoor temperatures in the U.S. vary from -30°C to +40°C. Barn temperatures range from 10°C to 40°C.
 - 4.1.3. The relationship between standard mass concentrations and actual concentrations also depends on air pressure. Atmospheric pressure varies from 88,000 to 104,000 Pa, or up to a 13% difference from standard pressure. Pressure can change geographically by 1000 Pa in a few hundred km.
 - 4.1.3.1. The static pressure differential between the inside and outside of mechanically-ventilated barns ranges from 0 to 80 Pa, or 0 to 0.08% of standard atmospheric pressure. Thus, the maximum variation of building static pressure relative to atmospheric pressure is typically only 0.01%, and is considered negligible.

- 4.1.4. EPA Method 4 specifies that PM concentrations should be reported based on dry air. The percentage of moisture in the exhaust air from livestock and poultry barns is typically 1-3%.

5. Personnel Qualifications

- 5.1. Most of these procedures assume a familiarity with the use of personal computers, general computer application software (e.g. MS Office), and the data analysis software CAPECAB (SOP B6).
- 5.2. Personnel should understand the Ideal Gas Law and its applications in concentration measurements. Personnel should also have basic knowledge of AFO (Animal feeding operation) emission measurement and expected ranges of measured variables.
- 5.3. Only personnel who are explicitly authorized to access the research data will be allowed to modify or write to the files.

6. Procedures

- 6.1. Data reporting
6.1.1. Concentrations

- 6.1.1.1. Report concentrations using the units listed in Table 1.

Table 1. Units of gas and PM concentrations at different conditions.

Concentrations	Gas, in ppbv	Gas, in ppmv	PM
Volumetric	Ppb	ppm	---
Mass (actual)	$\mu\text{g}/\text{m}^3$	mg/m^3	$\mu\text{g}/\text{m}^3$
Mass (standard)	$\mu\text{g}/\text{sm}^3$	mg/sm^3	$\mu\text{g}/\text{sm}^3$
Mass (dry standard)	$\mu\text{g}/\text{dsm}^3$	mg/dsm^3	$\mu\text{g}/\text{dsm}^3$

- 6.1.1.2. Report concentrations with a maximum of three (3) significant digits (e.g. 1.22, 20.2, 1220, etc.).

- 6.1.1.2.1. Round concentrations over 1000 ppm to the nearest 10 ppm (e.g. 2050 ppm, 5010 ppm, etc.).

- 6.1.1.2.2. Include the decimal for a concentration less than 100 ppm (e.g. report 20 ppm as 20.0 ppm), but drop it if over 100 (e.g. 122 ppm, not 122.0 ppm).

- 6.1.1.2.3. Supersede this rule in any case where it would imply greater accuracy than the instruments are capable of achieving. For example, ambient concentrations might be less than 1 ppm (e.g. 0.322 ppm). However, this would give the impression that the measurement is accurate to 1 ppb. If this is not the case, report the measurement as 0.32 or 0.3 ppm, depending on the sensitivity of the particular analyzer.

- 6.1.2. Emission rates

- 6.1.2.1. Report emission rates using the units listed in Table 2.

- 6.1.2.2. Report emission rates with a maximum of three (3) significant digits.

- 6.1.2.3. Emissions on a per-second basis are used to present diurnal emissions (hourly, per min, etc.). Baseline emissions are typically given on a daily or annual basis.

Table 2. Units of gas and PM emission rates.

Emission rate	Gas measured in ppbv	Gas measured in ppmv	PM
Per barn	kg/d mg/s kg/yr	kg/d g/s kg/yr	kg/d g/s kg/yr
Per unit floor area	$\mu\text{g/s-m}^2$ $\mu\text{g/d-m}^2$ g/yr-m^2	mg/s-m^2 g/d-m^2 g/yr-m^2	mg/s-m^2 g/d-m^2 g/yr-m^2
Per animal	$\mu\text{g/s-hd}$ $\mu\text{g/d-hd}$ g/yr-hd	mg/s-hd g/d-hd g/yr-hd	mg/s-hd g/d-hd g/yr-hd
Per live mass (AU=500 kg)	$\mu\text{g/s-AU}$ mg/d-AU kg/yr-AU $\mu\text{g/h-kg}$	mg/s-AU kg/d-AU kg/yr-AU mg/h-kg	mg/s-AU kg/d-AU kg/yr-AU mg/h-kg

6.2. Graphs and tables

- 6.2.1. Graphs should follow a particular style as defined by a prominent journal (e.g. Transactions of ASABE), which defines a specific format (font, labels, etc.).
- 6.2.2. Use the variables and format presented in Appendix A of this SOP to present data.
- 6.2.3. Use the “CreateTable” function of CAPECAB software (SOP B6) to create the monthly tables.

6.3. Calculation methods

6.3.1. Standardizing gas concentrations

- 6.3.1.1. Use the Ideal Gas Law ($PV = nRT$), expanded as in Equation 1, to convert volumetric gas concentrations to actual mass concentrations:

$$C = \frac{P \cdot c \cdot M}{R \cdot (273 + T_o)} \quad (1)$$

Where:

- C Actual mass concentration of gas (mg/m^3 or $\mu\text{g/m}^3$)
- P Atmospheric pressure at sampling location (atm)
- c Volumetric concentration of gas (ppm or ppb)
- M Molecular weight of gas (g/mol)
- R Universal Gas Constant (0.08206 L-atm/mol-°K)
- T_o Temperature at sampling location (°C)

- 6.3.1.2. Use the Ideal Gas Law to standardize the volumetric gas concentration of any pollutant (PM, gas) to the standard mass concentration on a moist-air basis (C'), as in Equation 2:

$$C' = \frac{P' \cdot c \cdot M}{R \cdot (273 + T')} \quad (2)$$

Where:

C' Standard mass concentration, moist-air basis (mg/sm³ or µg/sm³)

P' Standard pressure (1 atm)

T' Standard temperature (20°C)

6.3.1.3. Calculate the dry standard concentration according to Equation 3:

$$C'' = \frac{C'}{(1 - W)} \quad (3)$$

Where:

C'' Dry standard mass concentration dry basis (mg/dsm³ or µg/dsm³)

W Humidity ratio (See Appendix D)

6.4. Standardizing airflow rates

6.4.1. Equation 4 standardizes actual airflow (m³/s) to the moist standard airflow (sm³/s):

$$Q' = \frac{Q_o \cdot P \cdot (273.15 + T')}{P' \cdot (273 + T_o)} \quad (4)$$

Where:

Q' Moist standard airflow (sm³/s)

Q_o Outlet airflow rate at T (m³/s)

P Pressure at sampling location (atm)

T' Standard temperature (20°C)

P' Standard pressure (1 atm)

T_o Temperature at sampling location (°C)

6.4.2. Equation 5 standardizes actual airflow (m³/s) to dry standard airflow (dsm³/s):

$$Q'' = (1 - W) \cdot Q' = (1 - W) \cdot \frac{Q_o \cdot P \cdot (273.15 + T')}{P' \cdot (273 + T_o)} \quad (5)$$

Where:

Q'' Dry standard airflow (dsm³/s)

W Humidity ratio (See Appendix D)

6.5. Gas concentration corrections

6.5.1. The sensitivity of a gas analyzer may drift over time, introducing errors into the measurement. Correct these errors by using the precision check results (SOP G9) before and after the measurement, and by assuming that the drift is linear between the two precision checks. Fig. 1 illustrates how this correction works.

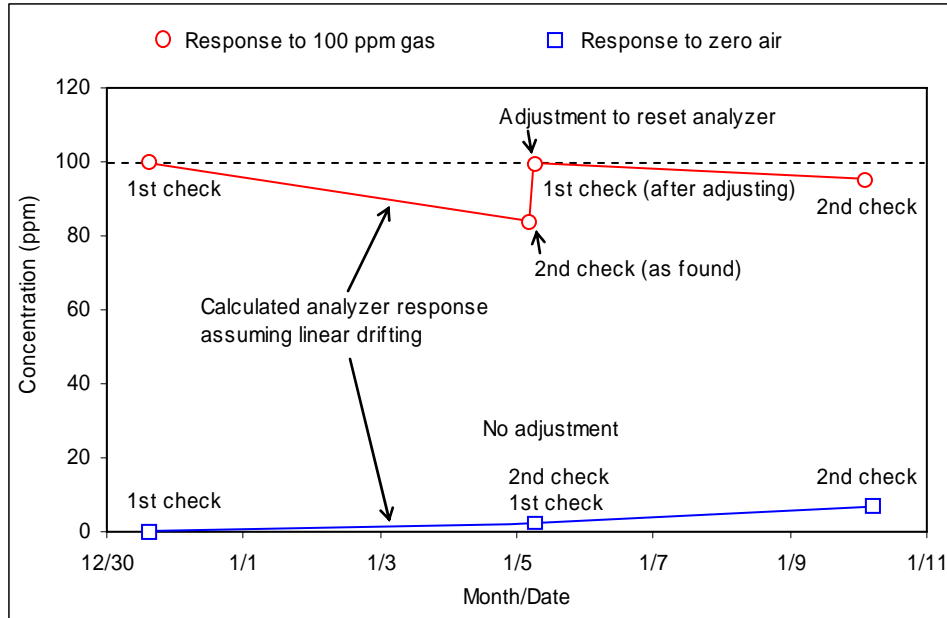


Figure 1. Linearly interpolated analyzer responses between the 1st and 2nd checks are used for correcting the data between the two checks.

6.5.2. The equations to correct gas concentrations based on two precision checks and linear interpolation are as follows:

$$c_m = Y_m - Xz_m - \left[(Xs_m - Xz_m) - (c_s - c_z) \right] \frac{(Y_m - Xz_m)}{(Xs_m - Xz_m)} \quad (6)$$

$$Xz_m = Xz_1 + (Xz_2 - Xz_1) \frac{(t_m - tz_1)}{(tz_2 - tz_1)} \quad (7)$$

$$Xs_m = Xs_1 + (Xs_2 - Xs_1) \frac{(t_m - ts_1)}{(ts_2 - ts_1)} \quad (8)$$

Where:

c	Corrected concentration measured at times between two precision checks (ppm or ppb)
c_s	Span gas concentration (ppm or ppb)
c_z	Zero air concentration (ppm or ppb)
t	Time of measurement
t_s	Time of span check
t_z	Time of zero check
X_s	PC as-found reading at span gas check
X_z	PC as-found reading at zero check
Y	PC-recorded concentration measured at times between two precision checks
1	Time of first precision check (subscript)
2	Time of second precision check (subscript)
m	Time of measurement (subscript)

6.5.2.1. Units of concentrations can be volumetric (ppm or ppb) or mass (mg/m^3 or $\mu\text{g}/\text{m}^3$) as long as only a single unit is used in the calculation.

6.6. Gas emission calculations

6.6.1. Calculate the net emission rate (i.e. building generation rate) by subtracting the inlet mass flow from outlet emission.

6.6.2. Two assumptions are implicit in the following discussion of emission rate calculations:

6.6.2.1. The inlet dry standard mass airflow rate is equal to the sum of all outlet dry standard mass airflow rates.

6.6.2.2. All gases obey the Ideal Gas Law.

6.6.3. The calculation of emission with a single ventilation exhaust sampling location is:

$$E = Q_o \cdot \frac{P_o \cdot M}{R \cdot (273 + T_o)} \cdot (c_o - c_i) \quad (9)$$

Where:

E	Barn emission rate (mg/s or $\mu\text{g}/\text{s}$)
Q_o	Barn outlet moist airflow rate at T_o (m^3/s)
P_o	Pressure at the exhaust sampling location (atm)
M	Gas molecular weight (g/mol)
R	Universal Gas Constant ($0.08206 \text{ L-atm}/\text{mol}\cdot^\circ\text{K}$)
T_o	Temperature at the exhaust sampling location ($^\circ\text{C}$)
c_o	Exhaust air concentration (ppm or ppb)
c_i	Ambient or ventilation air inlet concentration (ppm or ppb)

The derivation of Equation 9 is detailed in Appendix B.

6.6.3.1. Do not base emission calculations on dry standard concentration C_o'' and dry standard airflow Q_o'' . The reason is related to the fact that while moist standard incoming and outgoing flow rates are equal, the dry standard incoming and

outgoing flow rates are not equal, because moisture is added to the ventilation air in the barn.

- 6.6.4. Equation 9 can be used for each emission stream, since c_i (the inlet air concentration) is common to each stream. The barn emission rate with n sampling locations (corresponding to n ventilation exhausts) is:

$$E = \sum_{k=1}^n \left[Q_{o,k} \cdot (C_{o,k} - C_i) \right] \quad (10)$$

Where:

E	Gas emission rate from the barn (mg/s or $\mu\text{g/s}$)
$C_{o,k}$	Mass concentration at ventilation exhaust location k (mg/m^3 or $\mu\text{g/m}^3$)
C_i	Mass concentration in incoming ventilation air (mg/m^3 or $\mu\text{g/m}^3$)
$Q_{o,k}$	Ventilation rate at ventilation exhaust location k (m^3/s)

6.7. PM emission calculations

- 6.7.1. Use Equation 11 to calculate PM emission with a single ventilation exhaust sampling location.

$$E = \left\{ \left[Q_o \cdot P_o \cdot \frac{(273+25)}{(273+T_o)} \right] \cdot (C_o^* - C_i^*) \right\} \quad (11)$$

Where:

E	Net PM emission rate ($\mu\text{g/s}$)
Q_o	Exhaust airflow rate at T_o (m^3/s)
P_o	Pressure of exhaust air (atm)
C_o^*	PM concentration of exhaust air ($\mu\text{g/m}^3$)
C_i^*	Ambient PM concentration ($\mu\text{g/m}^3$)
T_o	Temperature of exhaust air ($^{\circ}\text{C}$)

The derivation of Equation 11 is detailed in Appendix C.

- 6.7.2. Also use Equation 11 for multiple emission stream emission calculation. However, if there is only one PM sampling location, substitute Q_o with the total barn ventilation rate, and Equation 11 will then yield the barn PM emission rate.
- 6.8. Concentration data extraction, averaging and interpolation
- 6.8.1. Gas concentration extraction and averaging
- 6.8.1.1. Gas concentrations of each SLG are generally measured continuously during a 10-min sampling period. Ambient air generally has a 30-min sampling period. Consult the QAPP to verify these sampling periods.
- 6.8.1.2. Depending on the response time and condition of different gas analyzers, each type of gas requires several min of pre-equilibrium data to be excluded from valid data sets for period mean calculation.

6.8.1.2.1. Some examples of the equilibrium time for several common analyzers are:

Ammonia (TEI 17C)	7 min invalid, 3 min valid
Hydrogen sulfide (TEI 450C)	5 min invalid, 5 min valid
Carbon dioxide (MSA 3600)	3 min invalid, 7 min valid

6.8.1.2.2. Observe analyzer traces during sampling and during precision checks to verify the equilibration period of each analyzer. The equilibration time for the ambient measurement data will be longer than the above-stated durations, because some analyzers require more time to reach equilibrium when switching from measuring high (exhaust location) to low (ambient) concentrations.

6.8.1.3. Pre-equilibration gas concentration readings during each sampling period are automatically flagged as invalid by the CAPECAB data processing software (SOP B6).

6.8.1.4. Assuming the data recording interval is 60 s, each valid reading is an actual minute average. Average the valid readings to obtain the sampling period mean.

6.8.2. Gas concentration interpolation

6.8.2.1. Estimate gas concentrations in the intervals between valid readings by linearly interpolating the means of two or three groups of valid readings.

6.8.2.2. The interpolation function utilizes the averages of the last x min of the most recent previous set of valid data, and of the first x min of the next set of valid data.

6.8.2.2.1. The value of x depends on the variance of the gas concentration

6.8.2.2.2. Interpolate to the midpoint of the interval with the x readings.

6.8.2.3. The maximum interval of missing data that should be interpolated will be project-specific, depending on the data quality objectives of the project. Consult the project QAPP for maximum allowable interpolation intervals.

7. Data and Records Management

7.1. Refer to SOP B5 for data and records management.

8. Quality Control and Quality Assurance

8.1. Completeness of data sets

8.1.1. Only calculate and report average daily means (ADM) for complete-data days (CDD), days in which over 70% of the data are valid.

8.1.1.1. This is necessary in order to avoid errors introduced into calculated average values due to partial-data days (*e.g.* only a few hours of valid data) that would result in biased time weights.

8.1.2. Similarly, only report hourly averages if more than 70% of the data during that hour are valid, and monthly averages only if more than 70% of the days are valid.

9. References

- 9.1. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 9.2. SOP B6. 2006. Data Processing Software (CAPECAB). Standard Operating Procedure B6. Purdue Ag Air Quality Lab
- 9.3. SOP G9. 2006. Precision Checks of Gas Analyzers. Standard Operating Procedure B6. Purdue Ag Air Quality Lab

Appendix B. Derivation of Gas Emission Equations

9.3.1. The calculation of emission with a single ventilation exhaust sampling location is:

$$E = (Q_o C_o - Q_i C_i) = (Q_o' C_o' - Q_i' C_i') \quad (B1)$$

Where:

E	Barn emission rate (mg/s or $\mu\text{g/s}$)
C_o	Mass concentration at the barn air inlet (mg/m^3 or $\mu\text{g/m}^3$)
C_i	Mass concentration at the barn air exhaust (mg/m^3 or $\mu\text{g/m}^3$)
C_i'	Standardized (STP) mass concentration at air inlet (mg/sm^3 or $\mu\text{g/sm}^3$)
C_o'	Standardized (STP) mass concentration at exhaust (mg/sm^3 or $\mu\text{g/sm}^3$)
Q_o	Barn outlet moist airflow rate at T_o (m^3/s)
Q_i	Barn inlet moist airflow rate at T_i (m^3/s)
Q_i'	Moist standard (STP) ventilation rate at air inlet (sm^3/s)
Q_o'	Moist standard (STP) ventilation rate at exhaust (sm^3/s)

9.3.2. Since there is conservation of mass, and $P_o/P_i \approx 1$, the airflow rate at T_o can be converted to airflow rate based on T_i , as follows:

$$Q_i = \left[Q_o \cdot \frac{P_o}{P_i} \cdot \frac{(273+T_i)}{(273+T_o)} \right] \approx \left[Q_o \cdot \frac{(273+T_i)}{(273+T_o)} \right] \quad (B2)$$

Where:

P_o	Pressure at the exhaust sampling location (atm)
P_i	Pressure of incoming air (atm)
T_o	Temperature at the exhaust sampling location ($^{\circ}\text{C}$)
T_i	Temperature of incoming air ($^{\circ}\text{C}$)

9.3.3. Combining Equations 1, B1, and B2, the net gas emission rate is:

$$E = \left\{ \left[Q_o \cdot c_o \cdot \frac{P_o \cdot M}{R \cdot (273+T_o)} \right] - \left[Q_o \cdot \frac{273+T_i}{(273+T_o)} \cdot c_i \cdot \frac{P_o \cdot M}{R \cdot (273+T_i)} \right] \right\} \quad (B3)$$

$$E = Q_o \cdot \frac{P_o \cdot M}{R \cdot (273 + T_o)} \cdot (c_o - c_i) \quad (\text{B4})$$

Where:

M	Gas molecular weight (g/mol)
c_o	Outlet (exhaust sampling location) gas concentration (ppm)
c_i	Inlet (ambient sampling location) gas concentration (ppm)
M	Gas molecular weight
R	Universal Gas Constant (0.08206 L-atm/mol-°K)

Appendix C. Correcting PM₁₀ Concentrations from the TEOM

The net PM emission rate is calculated as:

$$E = \left\{ \left[Q_o \cdot P_o \cdot \frac{(273+25)}{(273+T_o)} \right] \cdot (C_o^* - C_i^*) \right\} \quad (C1)$$

Where:

E	Net PM emission rate ($\mu\text{g/s}$)
Q_o	Exhaust airflow rate at T_o (m^3/s)
P_o	Pressure of exhaust air (atm)
C_o^*	PM concentration of exhaust air ($\mu\text{g}/\text{m}^3$)
C_i^*	Ambient PM concentration ($\mu\text{g}/\text{m}^3$)
T_o	Temperature of exhaust air ($^{\circ}\text{C}$)

Derivation of Equation C1

The default setting in the TEOM automatically converts the concentration C to C^* (based on 25°C , 1 atm). The TEOM converts the actual sample airflow rate Q_s to Q_s^* based on 25°C and 1 atm, and calculates the concentration C^* from the mass collected on the filter. However, C^* needs to be converted to C' at STP (20°C , 1 atm) for calculating emissions and to C'' for reporting emissions on a dry standard air basis. No adjustment for pressure is needed because the TEOM is already corrected to standard pressure (1 atm) internally. We know that:

$$C' = \frac{\Delta m_f}{t \cdot Q'_s} \quad (C2)$$

whereas the TEOM sampler output is:

$$C^* = \frac{\Delta m_f}{t \cdot Q_s^*} \quad (C3)$$

Where:

Δm_f	PM mass on the filter collected during sampling time t (μg)
C'	PM concentration at STP ($\mu\text{g}/\text{m}^3$)
C^*	PM concentration (reported by TEOM) at 1 atm and 25°C ($\mu\text{g}/\text{m}^3$)
t	Time of sampling during which Δm_f was collected (s)
Q'_s	TEOM sample airflow rate at STP (m^3/s)
Q_s^*	TEOM sample airflow rate at 1 atm and 25°C (m^3/s)

To get C' , we know

$$Q'_s = Q_s \cdot \frac{(273 + T')}{(273 + T^*)} \quad (C4)$$

Where:

T' Standard temperature (20°C)

T^* Temperature basis of TEOM reported concentrations (25°C)

And equation C2 becomes:

$$C' = \frac{\Delta m_f}{t \cdot Q'_s} = \frac{\Delta m_f}{t \cdot Q_s \cdot \frac{(273 + T')}{(273 + T^*)}} = \frac{\Delta m_f}{t \cdot Q_s} \cdot \frac{(273 + T^*)}{(273 + T')} \quad (C5)$$

Substituting from equation C3 yields

$$C' = C^* \cdot \frac{(273 + T^*)}{(273 + T')} \quad (C6)$$

The reportable concentration C'' is based on dry air:

$$C'' = \frac{C'}{(1 - W)} \quad (C7)$$

The barn airflow rate is used as follows in the calculation of net emission rate:

$$E = (Q_o C_o - Q_i C_i) = (Q'_o C'_o - Q'_i C'_i) \quad (C8)$$

Note that:

$$Q'_o = Q_o \cdot \frac{P_o}{P'} \cdot \frac{(273 + T')}{(273 + T_i)} \quad (C9)$$

$$C'_o = C_o \cdot \frac{(273 + T_o^*)}{(273 + T')} \quad (C10)$$

$$Q'_i = Q_i \cdot \frac{P_i}{P'} \cdot \frac{(273 + T')}{(273 + T_o)} \quad (C11)$$

$$C_i' = C_i^* \cdot \frac{(273+T_i^*)}{(273+T')} \quad (C12)$$

Substituting Equations C9 to C12 into Equation C8, yields:

$$E = \left\{ \left[Q_o \cdot \frac{P_o}{P'} \cdot \frac{(273+T')}{(273+T_o)} \cdot C_o^* \cdot \frac{(273+T_o^*)}{(273+T')} \right] - \left[Q_i \cdot \frac{P_i}{P'} \cdot \frac{(273+T')}{(273+T_i)} \cdot C_i^* \cdot \frac{(273+T_i^*)}{(273+T')} \right] \right\} \quad (D13)$$

$$E = \left\{ \left[Q_o \cdot \frac{P_o}{P'} \cdot \frac{(273+T_o^*)}{(273+T_o)} \cdot C_o^* \right] - \left[Q_i \cdot \frac{P_i}{P'} \cdot \frac{(273+T_i^*)}{(273+T_i)} \cdot C_i^* \right] \right\} \quad (D14)$$

9.3.4. Since there is conservation of mass, and $P_o/P_i \approx 1$, the airflow rate at T_o can be converted to airflow rate based on T_i , as follows:

$$Q_i = \left[Q_o \cdot \frac{P_o}{P_i} \cdot \frac{(273+T_i)}{(273+T_o)} \right] \approx \left[Q_o \cdot \frac{(273+T_i)}{(273+T_o)} \right] \quad (C15)$$

Where:

P_o	Pressure at the exhaust sampling location (atm)
P_i	Pressure of incoming air (atm)
T_o	Temperature at the exhaust sampling location (°C)
T_i	Temperature of incoming air (°C)

Substituting Equation C15 into Equation C14, and simplifying, yields:

$$E = \left\{ \left[Q_o \cdot \frac{P_o}{P'} \cdot \frac{(273+T_o^*)}{(273+T_o)} \right] \cdot (C_o^* - C_i^*) \right\} \quad (C16)$$

Since $P' = 1$ atm, and $T_o^* = 25^\circ\text{C}$, then:

$$E = \left\{ \left[Q_o \cdot P_o \cdot \frac{(273+25)}{(273+T_o)} \right] \cdot (C_o^* - C_i^*) \right\} \quad (C1)$$

Appendix D. Humidity Ratio Calculation

The humidity ratio (W) is calculated with Equation D1:

$$W = 0.62198 \cdot \frac{e^{f(T)} \phi}{P_{act} - e^{f(T)} \phi} \quad (D1)$$

Where:

W Humidity ratio (kg/kg)

P Pressure at the sampling location (Pa)

ϕ Relative humidity, decimal

Note that, for cases where $T < 273.15^\circ\text{K}$:

$$f(T) = \frac{C_1}{T} + C_2 + C_3 T + C_4 T^2 + C_5 T^3 + C_6 T^4 + C_7 \ln T \quad (D2)$$

And, for cases where $T > 273.16^\circ\text{K}$:

$$f(T) = \frac{C_8}{T} + C_9 + C_{10} T + C_{11} T^2 + C_{12} T^3 + C_{13} \ln T \quad (D3)$$

In Equations D2 and D3:

$C_1 = -5.675 \times 10^3$	$C_2 = 6.392$	$C_3 = -9.678 \times 10^{-3}$	$C_4 = 6.222 \times 10^{-7}$
$C_5 = 2.075 \times 10^{-9}$	$C_6 = -9.484 \times 10^{-13}$	$C_7 = 4.163$	$C_8 = -5.800 \times 10^3$
$C_9 = 1.391$	$C_{10} = -4.864 \times 10^{-2}$	$C_{11} = 4.176 \times 10^{-5}$	$C_{12} = -1.445 \times 10^{-8}$
$C_{13} = 6.545$			

DATA MANAGEMENT FOR BARNS
Standard Operating Procedure (SOP) B5

DATA MANAGEMENT FOR BARNS
Standard Operating Procedure (SOP) B5

Prepared by
Ji-Qin Ni and Juan Carlos Ramirez

Reviewed by
Albert J. Heber and Bill W. Bogan

Effective Date: November 6, 2006

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)
Agricultural and Biological Engineering, Purdue University
West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions	3
4. Personnel Qualifications and Responsibilities	3
5. Procedures	4
6. Quality Control and Quality Assurance	7
7. References.....	8

1. Scope and Applicability

- 1.1. The objective of data management is to ensure data security, easy data identification and retrieval, and efficient publication.
- 1.2. This SOP applies to the management of air emission data collected at commercial livestock barns. It applies to all affiliated personnel who conduct research, assist in the research, or perform duties that bring them in contact with the following data:
 - 1.2.1. Measurement variables that are monitored by the data acquisition computer.
 - 1.2.2. Data from off-line sample analysis, including manure characteristics and particulate matter weights, by various laboratories.
 - 1.2.3. Data from the nearest weather station.
 - 1.2.4. Livestock characteristics obtained from the producer.
 - 1.2.5. Field test notes and visual observations.
 - 1.2.6. Data analyzed to calculate air emission rates.
 - 1.2.7. Farm and barn design and operational characteristics.

2. Summary of Method

Electronic files are classified according to their importance and recoverability. All project-related files in the on-site computer are arranged in a single folder with a default structure of subfolders. An administrative server on campus stores data from on-site computers and data after processing. Files in the on-site computers are backed-up regularly. Acquired 24-h measurement data are emailed to the administrative server and site PI daily. Field notes and other electronic files (e.g. digital photos) are emailed to recipients daily when they are updated. The project data files are kept for at least six years.

3. Definitions

- | | |
|----------------------|---|
| 3.1. ABE | Agricultural and Biological Engineering. |
| 3.2. AirDAC | Air Data Acquisition and Control program. |
| 3.3. CAPECAB | Calculation of Air Pollutant Emissions from Confined Animal Buildings, a custom computer program. |
| 3.4. Data recipients | Individuals designated to receive emailed data. |
| 3.5. Site PI | Site principal investigator. |
| 3.6. SMP | Site monitoring plan. |

4. Personnel Qualifications and Responsibilities

- 4.1. Most of these procedures assume a familiarity with the use of personal computers, general computer application software, e.g. MS Office, and the data acquisition and control software, AirDAC (SOP B2) or the data analysis software CAPECAB (SOP B6).
- 4.2. The site PI will follow the procedures of data recording, organization, and safeguards and should ensure that all members of the research team (including appropriate administrative

- personnel) understand and follow the procedures for the site.
- 4.3. Only those individuals explicitly authorized to access the research data will be allowed to write to the files. The person creating the daily field notes will be identified in the notes.
 - 4.4. Those authorized to access research data will use strong passwords that are difficult to crack. Passwords will not be written down or shared with others. Passwords will be changed on a regular basis (every two months at minimum).
 - 4.5. The authorized personnel are site PI, site engineer, project managers, and data analysts.
 - 4.5.1. The site PI and site engineer have keys to the on-farm instrument shelter and access to the site computer.
 - 4.5.2. The site PI and site engineer have the password to remotely connect to the site computer.
 - 4.5.3. The site PI and site engineer have read/write access to the site data.
 - 4.5.4. The project managers and data analysts have passwords to the computers that are connected to the administrative server.
 - 4.5.5. The project managers and data analysts have read/write access to the administrative server data.
 - 4.5.6. No one is authorized to change original raw data files (Sections 6.4 and 6.5).

5. Procedures

- 5.1. Classification of electronic files
 - 5.1.1. Classify all electronic files according to the following four categories:
 - 5.1.1.1. Category 1. Research information and data. These files, if lost, cannot be rebuilt or recovered.
 - 5.1.1.1.1. Measurement data including data logged by instruments with internal data storage
 - 5.1.1.1.2. Data acquisition and control configuration data
 - 5.1.1.1.3. Calibration data recorded by AirDAC, electronic calibration forms
 - 5.1.1.1.4. Field notes
 - 5.1.1.1.5. Email messages
 - 5.1.1.1.6. Image files (digital photos or computer screen shots)
 - 5.1.1.1.7. Farm production data (excluding copies obtained from farm management)
 - 5.1.1.2. Category 2. Project-specific configurations that could be rebuilt or recovered, if lost.
 - 5.1.1.2.1. Hardware configuration files (e.g. FieldPoint configuration file *.iak)
 - 5.1.1.2.2. Configurations of firewalls, virus protection, etc.
 - 5.1.1.2.3. Email accounts, address books, message rules
 - 5.1.1.2.4. Favorite lists
 - 5.1.1.3. Category 3. Project-specific software and documents
 - 5.1.1.3.1. Files provided by vendors such as instrument manuals and quotations
 - 5.1.1.3.2. Data acquisition and control software (e.g. C:\MCC)
 - 5.1.1.3.3. Instrument communication software
 - 5.1.1.4. Category 4. Operation system and general PC software
 - 5.1.1.4.1. Windows
 - 5.1.1.4.2. MS Office

5.1.1.4.3. pcAnywhere

5.1.1.4.4. Other software

5.2. File organization

5.2.1. In the on-site data acquisition computer, arrange all project-specific files in a single default folder called YYnX where YY is a two-letter abbreviation for the state and n is a number and X is a letter. For example, a site in Indian would be identified as NC1B. Organize the subfolders of YYnX in the C drive as shown in Fig. 1:

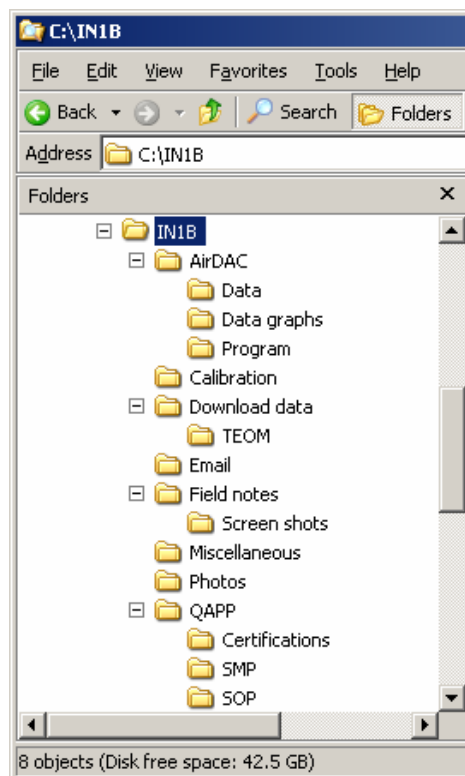


Figure 1. An example of default project folder structure in the on-site computer.

5.2.1.1. AirDAC. Contains air data acquisition and control.

5.2.1.1.1. Data. Stores raw text data collected by the data acquisition program.

5.2.1.1.2. Data graphs. Contains daily plots of raw data.

5.2.1.1.3. Program. Contains AirDAC program and configuration files for AirDAC.

5.2.1.2. Calibration. Contains calibration sheets, gas cylinder logs, control charts, and other records.

5.2.1.3. Download data. Contains data downloaded from instrument internal data loggers.

5.2.1.4. Email. Contains email messages and attachments. Name the email account YYnX@zzz.zzz where zzz.zzz is the internet service provider's domain. Outlook Express is the preferred email program.

5.2.1.5. Field notes. Contains typed logs of activities and observations.

5.2.1.5.1. Screen shots. Contains interesting trend histories consisting of 1-s readings.

5.2.1.6. Miscellaneous. Contains site equipment inventory including gas cylinders, producer supplied information (e.g. Producer Event Forms), etc.

- 5.2.1.7. Photos. Stores digital pictures of field setup and observations.
- 5.2.1.8. QAPP. Saves the latest copy of the Quality Assurance Project Plan.
 - 5.2.1.8.1. Certifications
 - 5.2.1.8.2. SMP. Site monitoring plan
 - 5.2.1.8.3. SOP. Standard operating procedures (SOP).
- 5.2.2. The administrative server on campus contains the following information:
 - 5.2.2.1. YYnX. A copy of the files received from the on-site DAC computers.
 - 5.2.2.2. Data emailed from YYnX and automatically saved in this folder.
 - 5.2.2.3. CAPECAB data and configuration files.
 - 5.2.2.4. Excel files of data exported from CAPECAB during processing.
 - 5.2.2.5. PowerPoint slides for presentations
 - 5.2.2.6. Drawings of barns and equipment
 - 5.2.2.7. Data tables (see SOP B4) for reports and manuscripts.
 - 5.2.2.8. Reports
 - 5.2.2.9. Manuscripts
- 5.3. Data safety and security
 - 5.3.1. The on-site computer will be equipped with two parallel hard drives, so that all files will be backed up to the second drive. Execute a progressive back-up of the project folder (YYnX), from the primary hard drive of the on-site computer to the secondary hard drive, such that all files modified in a given day will be backed up at the conclusion of that day. This will be done automatically on a daily basis.
 - 5.3.2. Select data recipients and configure the AirDAC (SOP B2) to email data to. The data recipients include Site PI, Site post-doc, and the administrative server (odorair@purdue.edu).
 - 5.3.3. Program the AirDAC to email the 24-h data files to the data recipients defined in Section (5.3.2) at midnight (SOP B2).
 - 5.3.4. Send calibration data and control charts to recipients defined in Section (5.3.2) after calibrations or precision checks.
 - 5.3.5. Email field notes to recipients defined in Section (5.3.2) at the end of each work day during which the field notes are edited or added to (i.e. any day that work is done on-site), before leaving the site or logging out of a remote connection to the on site computer. The individual who creates the daily field notes (whose name is recorded in the notes as per Section 4.3) will be contacted in any case of missing data and the site PI will also be contacted or notified so corrective action will be taken.
 - 5.3.5.1. Any photos which need to be included into the field notes will be taken with a digital camera (kept on site), transferred to the computer, and e-mailed along with the electronic field notes for the corresponding day.
 - 5.3.5.2. Any paper drawings which need to be included into the field notes will be scanned (kept on site), transferred to the computer, and e-mailed along with the electronic field notes for the corresponding day.
 - 5.3.6. Email AirDAC configuration files to recipients after updating them.
 - 5.3.7. Back up the entire project folder YYnX, which include all the Category 1 files, from on-site computer onto an external backup device (e.g. hard drive, DVD, or CD) every 2 wks or when there are changes in Category 2 or 3 files.
 - 5.3.8. Keep the external backup device off-site for storage by the site PI or site post-doc.

- 5.3.9. Back up operating system to the secondary hard drive when there are significant changes, e.g. new project-related software installed or updated.
- 5.3.10. Back up the project administrative server files to the Purdue University Engineering Computing Network's backup system daily and automatically. The computer administrator of the ABE Dept., Purdue University has the authority to retrieve the backup data from the Purdue University backup system.
- 5.3.11. The passwords are updated the same day upon termination of a person's work with the project.
- 5.4. Management of producer-supplied data
 - 5.4.1. As described in SOP S1, producers are expected to keep a "Producer Event Form", which must document all production levels, changes in inventory, significant mortalities, manure removals, cleanings, and any other factors which would be expected to influence emissions.
 - 5.4.2. Collect Event Forms from the producer on a weekly basis.
 - 5.4.2.1. Keep hard copies of Event Forms on site in a bound notebook.
 - 5.4.2.2. Scan and save Event Forms to the on-site computer (See Section 5.3.5.2), and e-mail to the data recipients on a weekly basis.
- 5.5. Retention of data
 - 5.5.1. At the conclusion of the project, remove the primary hard disk (C drive) from the site computer and erase all files from the secondary hard disk so that the data can be protected if the site computer is to be used for other purposes.
 - 5.5.2. Keep the removed hard disk, producer-supplied data (Section 5.4), and other hard copies, along with the "off-site" backup device, for at least six (6) years by the site PI. The site PI has access and control of the files.
 - 5.5.3. Keep all project related files in the administrative server, including the files received from the site computers, for at least six (6) years by PAAQL, Purdue University. The PAAQL personnel involved in this project has access and control of these data.
 - 5.5.4. Establish digital signatures of data using LabVIEW and its DSC (Data Supervision and Control) module, so that it is compliant with FDA regulation 21 CFR Part 11.

6. Quality Control and Quality Assurance

- 6.1. Make and follow the schedule of file backups described in Section 5.3.
- 6.2. Record in the field notes any manual backup that has been performed. in the field notes in a worksheet called "Backup."
- 6.3. Designate a person from the authorized personnel (Section 4.5) to back up files.
- 6.4. Check field computer and the relevant software, and take immediate measures to solve the related problem if the daily data file email, or emails with other files are not received.
- 6.5. Back up all raw data in their original form.
- 6.6. If corrections and changes are needed in the created raw files, including the manually entered original data files (e.g. manure sampling locations), perform the following:
 - 6.6.1. Make a copy of the original file and keep the original file unchanged, including the file saving date and time.
 - 6.6.2. Work only on the copied file.
 - 6.6.3. Record in the new file who made the corrections/changes and when and why the data

were corrected/changed. For Excel files, make the record in a new worksheet "Notes." Records can also be made as comments in the relevant cells.

- 6.6.4. Rename the new file by adding or updating the date at the end of the original file name in the following format: mm-dd-yy, e.g. "B1 manure samples 01-09-06.xls" for an original file named "B1 manure samples.xls." Leading zeros in date and month can be omitted, e.g. 1-9-06.
- 6.7. Establish electronic audit trails of data using LabVIEW and its DSC (Data Supervision and Control) module, so that it is compliant with FDA regulation 21 CFR Part 11.

7. References

- 7.1. EPA (2001). Guidance for Preparing Standard Operating Procedures (SOPs) EPA QA/G-6, EPA, EPA/240/B-01/004, Available at <http://www.epa.gov/QUALITY/qs-docs/g6-final.pdf>. Accessed October, 2005.
- 7.2. EPA (2003). Standard Operating Procedure for (PM2.5) Database Operations, US EPA, Environmental Health and Safety Division. Available at <http://www.epa.gov/ttnamti1/files/ambient/pm25/spec/database.pdf>. Accessed on December 20, 2005.
- 7.3. IUPUI/Clarian Standard Operating Procedure for Data Management, v 05/05. Available at <http://www.iupui.edu/~respoly/human-sop/SOP%20-%20Data%20Management.htm>. Accessed on December 21, 2005.
- 7.4. SOP B2. 2005. Standard Operating Procedure for Data Acquisition and Control Software (AirDAC). Purdue Ag Air Quality Lab.
- 7.5. SOP B4. 2005. Standard Operating Procedure for Calculation and Reporting of Air Emissions. Purdue Ag Air Quality Lab.
- 7.6. SOP B6. 2005. Standard Operating Procedure for Data Processing Software (CAPECAB). Purdue Ag Air Quality Lab.

**DATA PROCESSING PROGRAM (CAPECAB)
Standard Operating Procedure (SOP) B6**

DATA PROCESSING PROGRAM (CAPECAB)
Standard Operating Procedure (SOP) B6

Prepared by

Teng-Teeh Lim and Erin L. Cortus

Reviewed by

Albert J. Heber and Bill W. Bogan

Effective Date: December 28, 2007

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability.....	3
2. Summary of Method.....	3
3. Definitions.....	4
4. Personnel Qualifications and Responsibilities.....	5
5. Equipment and Supplies	5
6. Procedures	5
7. Records Management	15
8. Quality Control and Quality Assurance	17
9. References	17
10. Contact Information	17

1. Scope and Applicability

- 1.1. This SOP applies specifically to the custom software, Calculation of Air Pollutant Emissions from Confined Animal Buildings (CAPECAB). The program was developed to assemble and review data, and final-process air emission measurement variables collected from confined animal feeding operations (CAFO).
- 1.2. The CAPECAB program was originally written in Visual Basic 6 programming language. The program language is being converted to Visual Basic.Net, resulting in different user interfaces, and slight variations to some procedures. However, the focus of this SOP will be the use of CAPECAB to generate the final air emission measurement variables. Hereafter, the original program will be referred to as the VB6 version, and the revised program will be referred to as the .NET version.
- 1.3. The program converts measurement variables mainly acquired and generated by the data acquisition and control software, AirDAC (SOP B2), into the CAPECAB database. Other data files, including animal and feed consumption information, weather station data, stand-alone measurement data, and data logged by instruments (TEOM, SOP P1) with internal data storage, can also be sorted and combined into the database.
- 1.4. This method concatenates and displays graphical and tabular data over any desired interval between 10 min to 60 d for review and verification.
- 1.5. Basic statistics calculated by CAPECAB for user-defined intervals of data include frequency, maximum, minimum, and mean.
- 1.6. This method utilizes binary files that are processed much faster than spreadsheet files.
- 1.7. Other important functions of the software are to exclude pre-equilibrium and invalid data, and conduct airflow and emission rate calculations.
- 1.8. The software can also be used to provide original and processed data in table format for report writing.

2. Summary of Method

The CAPECAB program consists of four application subprograms: 1. Convert; 2. Display; 3. Flagging; and 4. ImportExport. A flowchart (Fig. 1) shows the program's data processing procedures. Tabular data (15-s or 60-s averages) collected by AirDAC (SOP B2) are first converted using the "Convert" program into a binary database where each column of data from AirDAC (representing a single measurement) becomes a separate file. Separate files are created for each month of data collection. Each datum is associated with the time stamp (1-min interval), the sampling location as specified by the AirDAC, and a flag value. Users are allowed to create hundreds of files to store raw (from AirDAC), imported (e.g., Producer Event Form data), and calculated data values. The "Display" program is capable of presenting graphical and tabular data consisting of minute-data or means over any user-specified time interval (hourly, daily, monthly, etc). When reporting mean values, users will identify the data completeness percentage of the mean values. This function is set up to prevent reporting incomplete data to avoid bias. The "Flagging" program is used to inspect and assess data validity, using a graphical interface. For example, data can be manually or automatically flagged invalid based on field notes, user-specified ranges, ventilation-stage dependent measurement, or when the user is inspecting the data. The "ImportExport"

program is used to incorporate data from other sources, and to execute various calculations for data processing such as adjustment of gas concentration data (due to analyzer drifts) based on precision checks, extraction of equilibrium data, calculation of ventilation rates, and finally, calculation of emission rates. At the end of data processing, the calculated mean values are exported into tabular format using the “Display” program.

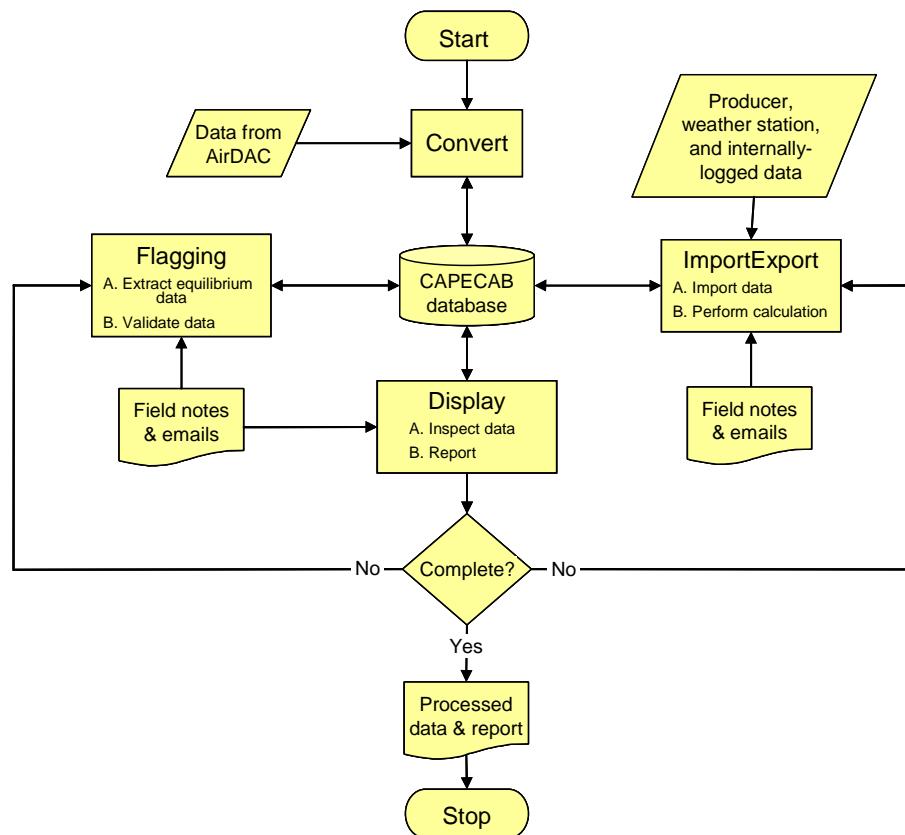


Figure 1. Data flow diagram of CAPECAB data processing.

3. Definitions

- | | | |
|------|---------|---|
| 3.1. | ADPP | Air Data Pre-Processing program, written in Visual Basic for Applications in MS Excel |
| 3.2. | CAFO | Confined animal feeding operations |
| 3.3. | AirDAC | Air Data Acquisition and Control program written in LabVIEW (SOP B2). |
| 3.4. | CAPECAB | Calculation of Air Pollutant Emissions from Confined Animal Buildings, a custom computer program |
| 3.5. | LabVIEW | Laboratory Virtual Instrumentation Engineering Workbench. A platform and development environment for a visual programming |

- language (a dataflow language), produced by National Instruments Corporation (Austin, TX)
- 3.6. LSAS Location-shared analyzers and sensors
 - 3.7. PAAQL Purdue Agricultural Air Quality Laboratory
 - 3.8. QAPP Quality Assurance Project Plan
 - 3.9. XXYB Site-specific reference where XX represents the state, Y represents the animal type, and B represents a barn source

4. 4. Personnel Qualifications and Responsibilities

- 4.1. Personnel are expected to have good computer skills, including Microsoft Excel.
- 4.2. All personnel involved in data processing must read and understand this SOP, and SOPs B2, B3, B4, and B5 to know the details of data acquisition and data processing.
- 4.3. Personnel involved in data inspection and processing are required to have basic knowledge of CAFO emission measurements, and the general ranges of measured variables. Personnel are expected to understand the data patterns (e.g. diurnal and seasonal changes), and the relationship among the data (e.g. effect of ventilation on concentrations), so that data can be inspected with expert judgment.
- 4.4. A training course by someone familiar with the CAPECAB software is recommended to supplement the information provided in this SOP.

5. Equipment and Supplies

- 5.1. Personal computer with minimum 1.5 GHz CPU speed, 512 MB RAM, and 4 GB free hard disk space, and installed with Windows XP and spreadsheet software
- 5.2. Calculation of Air Pollutant Emissions from Confined Animal Buildings (CAPECAB) software
- 5.3. Tabular data recorded or generated by AirDAC (SOPs B2, B3, and B5). These data files will be emailed to the appropriate user(s) daily, or can be transferred from the monitoring sites.
- 5.4. Analyzer precision check data files, which describes analyzer drifts (SOP B5), and will be used for gas concentration corrections
- 5.5. Field notes (in emails, or electronic copy in computer or network) and emails

6. Procedures

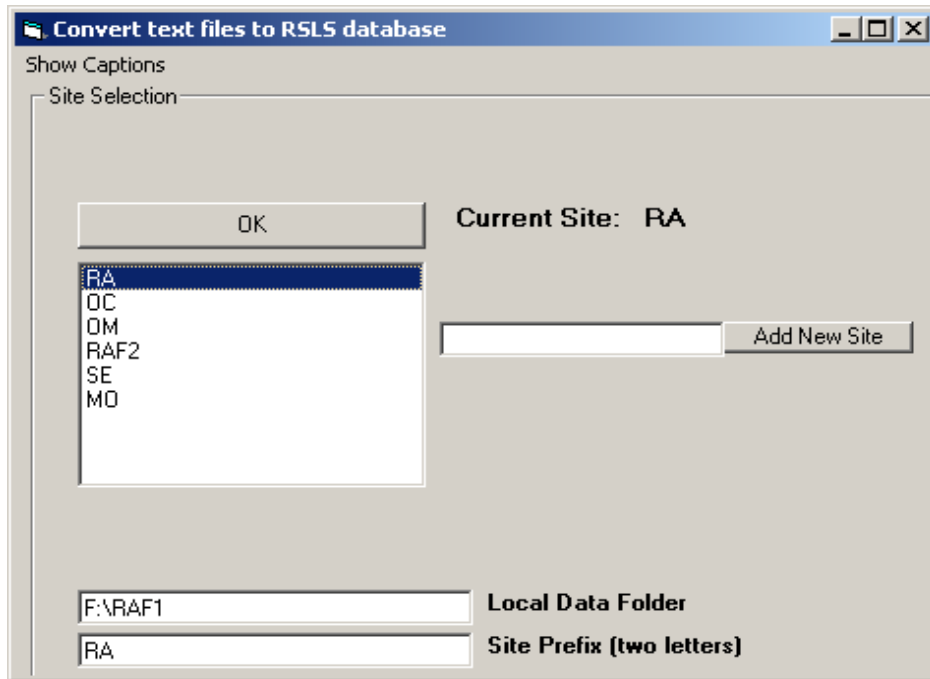
- 6.1. Set up CAPECAB configuration files.
 - 6.1.1. Prepare an array of configuration files for each monitoring site database group. The file type is “comma separated.” It is recommended to start from existing template files and incorporate the site-specific details into the files.
 - 6.1.2. Several important configuration files include ana_info_XXYB.csv, convert_info_XXYB.csv, and flagging_info_XXYB.csv. All of these files will be kept in the same site-specific folder, such as C:\Site_XXYB\CC6_XXYB\dif_XXYB, where XXYB is the site-specific CAPECAB database prefix name.

- 6.1.3. Modify the ana_info_XXYB file first to include details of the name, type and group, unit, and column number of each measurement variable.
- 6.1.3.1. Every measurement site has a unique data configuration. A good practice is to group the similar measurement variables, such as concentration data columns together, and assemble the other types accordingly.
- 6.1.3.2. Users can use either Flagging or Display function to check the database. Errors such as repeated column numbers, undefined columns, etc, will be indicated by individual windows when starting the program.
- 6.1.3.3. All of the configuration files can be created and edited using an MS Excel spreadsheet. Verify the line breaks in the configuration files using WordPad.
- 6.2. Convert tabular data into the CAPECAB database using the Convert program.
- 6.2.1. The main function of the Convert program is to assemble the AIRDAC data into the CAPECAB database. Setup the convert_info_XXYB file to indicate the column numbers for both the AirDAC tabular data and the CAPECAB database, Fig. 2. The CAPECAB database numbers essentially represent different files, but are referred to as columns for easier comparison with the original AirDAC data.
- 6.2.1.1. The heading row from an AirDAC data file can be copied and pasted in column 3 using MSEXcel function Paste Special – Transpose. Doing this helps to assign the appropriate CAPECAB column numbers to the AirDAC column data.
- 6.2.1.2. In the case of a column in the AirDAC data that is not used for a measurement (i.e. “Reserved”), assign a CAPECAB column number of -1.

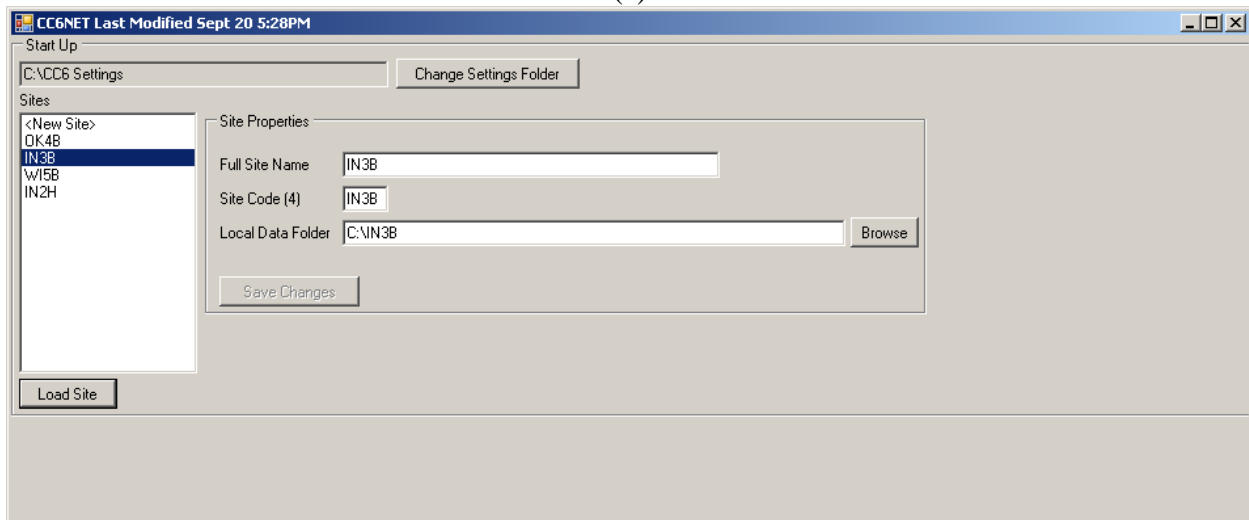
1	2	3	4	5	6	7
1		Year	Month	Day	Hour	Minute
2	*	TR	2007	1	1	0
3		-1	Date & time			
4		0	Smpl loc#			
5	*	1	Cal gas #	n	606	
6	*	2	Cal gas	ppm	607	
7	*	3	NH3	ppm	1000	
8	*	4	CH3OH	ppm	1500	
9	*	5	C2H6O	ppm	1600	
10	*	6	THC	ppm	1700	
11	*	7	CH4	ppm	1400	
12	*	8	WV	Tdew	302	
13	*	9	CH4	ppm	900	
14	*	10	NMH	ppm	1800	
15	*	11	H2S	ppb	1300	
16	*	12	SO2	ppb	1200	
17	*	13	CO2	ppm	1100	
18	*	14	Smpl P	Pa	603	
19	*	15	Smpl Q	L/m	601	
20	*	16	Smpl RH	%	200	
21	*	17	Smpl T	C	300	
22	*		end			
23						

Figure 2. Example of a convert_info_XXYB.csv file; column 2 indicates the source column numbers of AirDAC data file (starting with the date and time column as -1); column 5 indicates the destination column numbers in the CAPECAB database.

- 6.2.2. Open the Convert program (Fig. 3), and verify the information shown in the site selection window. If the program is used for the first time or has been re-installed, enter the corresponding local data folder and site prefix name, and save the information.



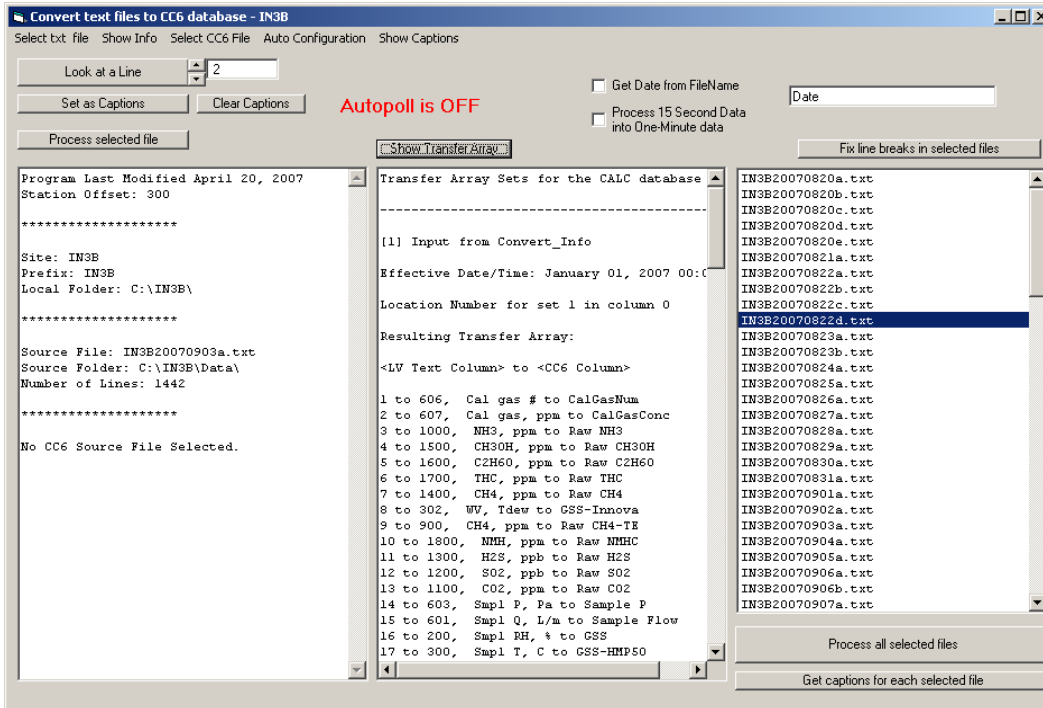
(a)



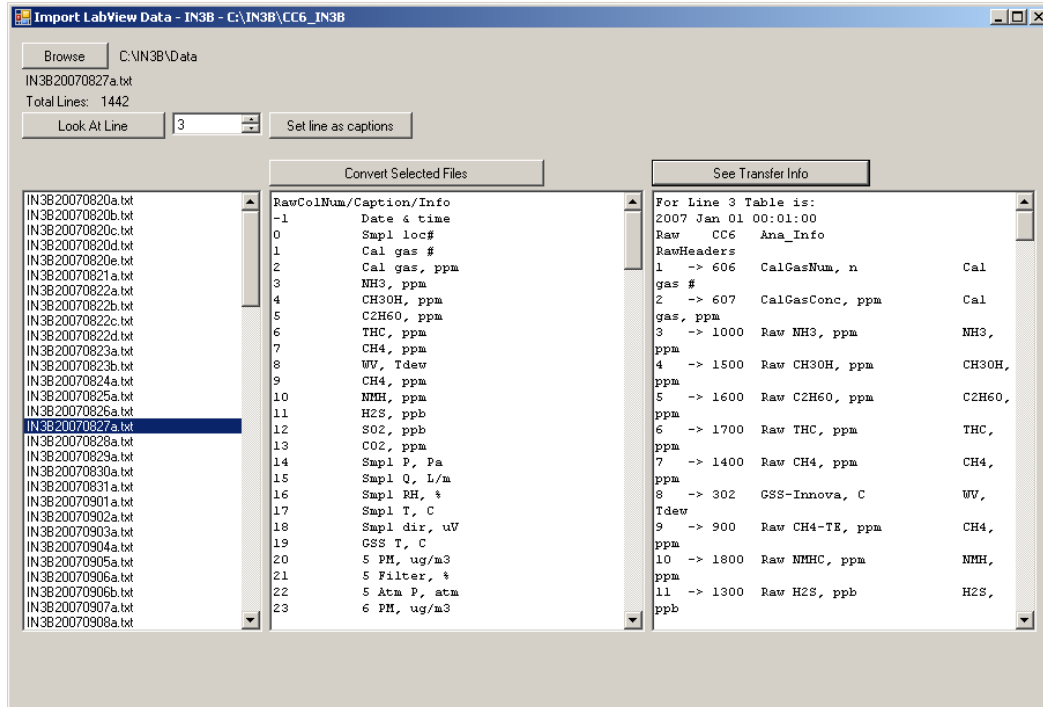
(b)

Figure 3. Example of a site-selection window of the Convert program using the VB6 version (a) and the .NET version (b).

- 6.2.2.1. When the Convert form (Fig. 4) appears, click “Select txt file” (VB6) or “Browse” (.NET) from the menu bar and open a folder containing the text file(s) to be converted. Select one of the text files and click “Open”. Select one of the text files in the convert program window. Change the number in the text box on the left hand side of the form to “2”, and click “Look at a Line”. Line 2 in the text files contains the AirDAC data column headings. Click “Set as Captions”, and then click “Show Transfer Array” (VB6) or “See Transfer Into” (.NET). The conversion can be verified by comparing the AirDAC (or LV for LabView) column headings to the column names for the destination files in the CAPECAB database (CC6 Column; names are assigned in the Ana_Info6_XXYB.csv file) in the middle window for the VB6 version, and the right-hand side window for the .NET version. Verify the data column numbers to be assembled. If there are any errors, review the Ana_Info6_XXYB and/or Convert_Info6_XXYB files.
- 6.2.2.2. Select the files to be assembled into the CAPECAB database and press “Process all selected files” (VB6) or “Convert Selected Files” (.NET) (Fig. 4).
- 6.2.2.3. Check the individual monthly CAPECAB folders to verify that the data are imported. The monthly files will be saved at the same folder level similar to the dif folder, which is C:\Site_XXYB\CC6_XXYB\. The monthly files will be named YYYY_MM_XXYB where YYYY is the year, MM is the month, and XXYB is the site prefix.
- 6.2.2.4. Verify the site-specific folder location and name of each sub-window.
- 6.3. Validating and extracting data using the Flagging program.
 - 6.3.1. The main purposes of the Flagging program are to inspect and validate data, and extract equilibrium data. The flag values in the CABECAB database range from 0 to 255. Any unflagged data is considered invalid and will not be reported, thus forcing users to inspect all data prior to analyzing it. Validated data have flag values of 0 or 100. All others are considered invalid.
 - 6.3.2. When data is first converted into the CAPECAB database, each datum is assigned a flag of 255, meaning “unflagged”. It is possible to initially assign a flag of 0 (Valid) to certain data columns.
 - 6.3.3. CAPECAB will suggest a flag to each data point, based on information in the Flagging_Info6_XXYB.csv file for LSAS variables, or by default, flags of 0 and 1 for perceptible and missing data, respectively.
 - 6.3.3.1. Select a measurement variable and a range of time to inspect and flag. A reasonable interval of time to inspect data is 1 or 2 d. Users can zoom into a small section of data to inspect, and only accept the valid flags if no error is found.
 - 6.3.3.2. Click “Show/Hide Minute Editor”. The top row of the Minute Editor Form will show the suggested flags (Fig. 5a). If these flags are acceptable, click “Transfer Flags” and the new flag values will appear for each data point (Fig. 5b).

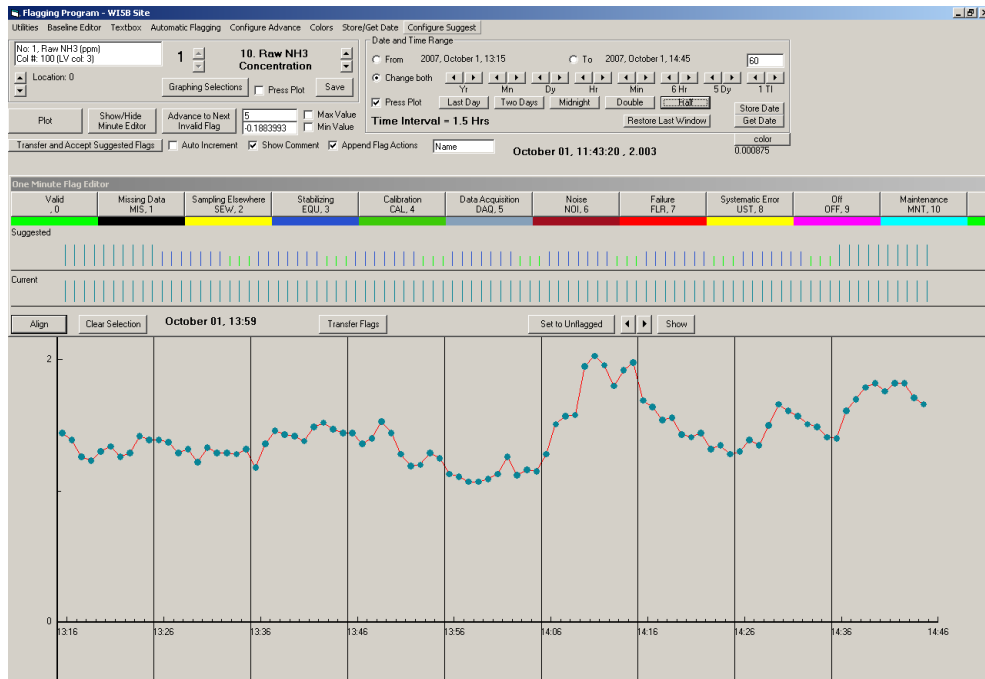


(a)

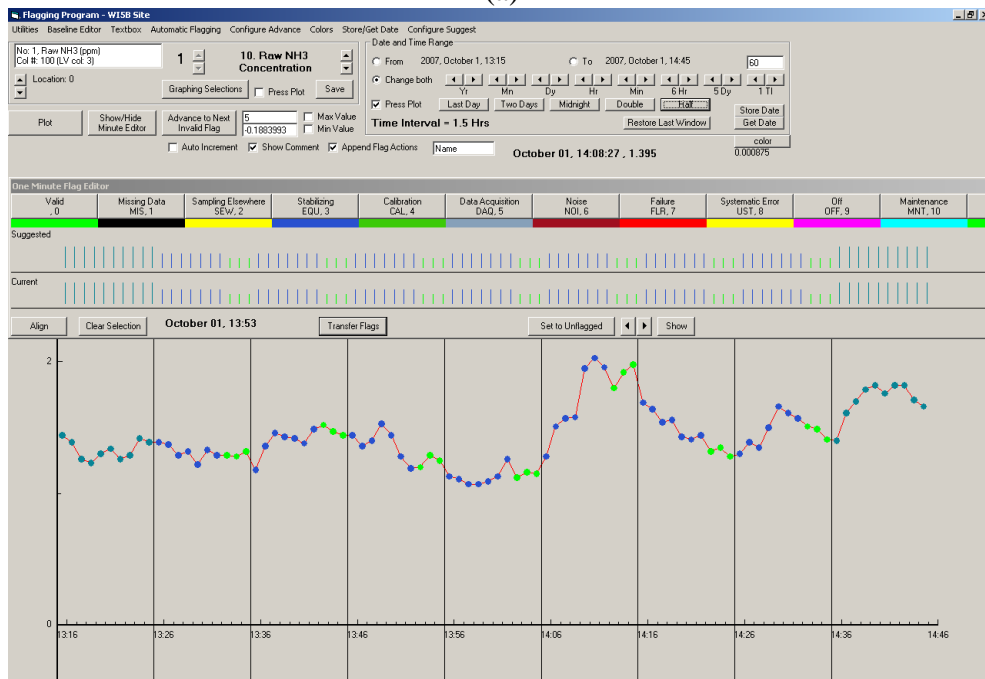


(b)

Figure 4. Example of a VB6 Convert program interface (a) and a .NET Convert program interface (b) with individual windows showing CAPECAB database folder information, column numbers, and data files to be assembled.

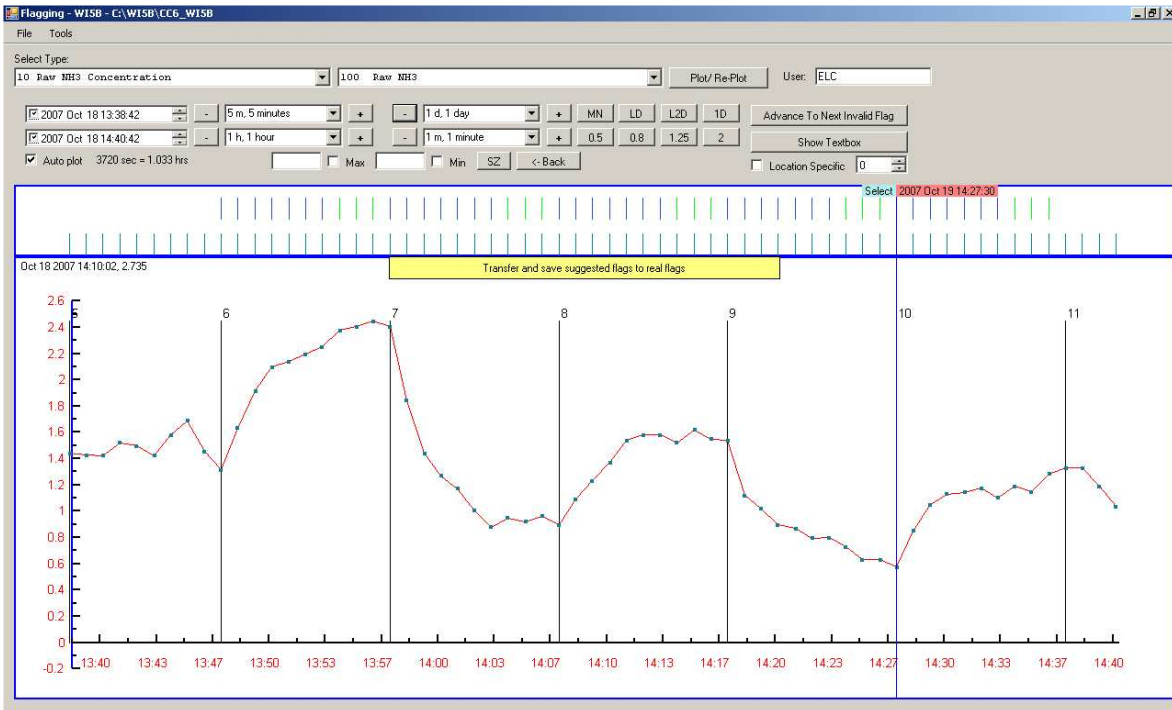


(a)

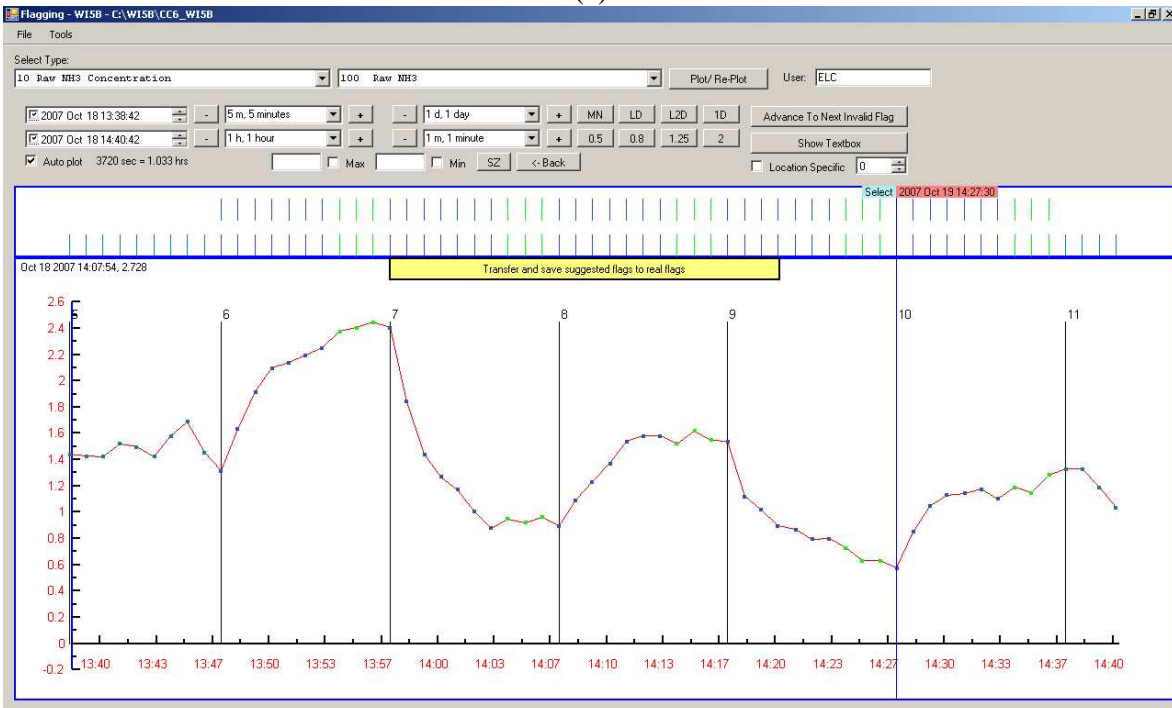


(b)

Figure 5. Example (VB6 version) of using the Flagging program with ammonia measurements. Unflagged data (a) can be flagged using the suggested flags in the One Minute Flag Editor (b). The first 7 min of sampling period are invalidated (blue dots) because the analyzer is stabilizing, while the last 3 min of sampling period are valid (green dots).



(c)



(d)

Figure 6 (cont). Example (.NET version) of using the Flagging program with ammonia measurements. As above, unflagged (c) and flagged (d) data are shown.

- 6.3.4. To manually flag data , click the “Show/Hide Minute Editor” button. Hold down the the left button of the mouse and drag a line across the desired range of data points. Click on the appropriate flag and add any appropriate comments in the text box on the upper left hand side of the form. Also, ensure that your name or initials are displayed in the text box in the middle of the form. Click “Save Edits to File” (Fig. 6). A record of manual flagging events containing the name of the user, the date and time the flag was made, and the flagged data information is stored in the flagevents.txt file in the dof_XXYB folder.

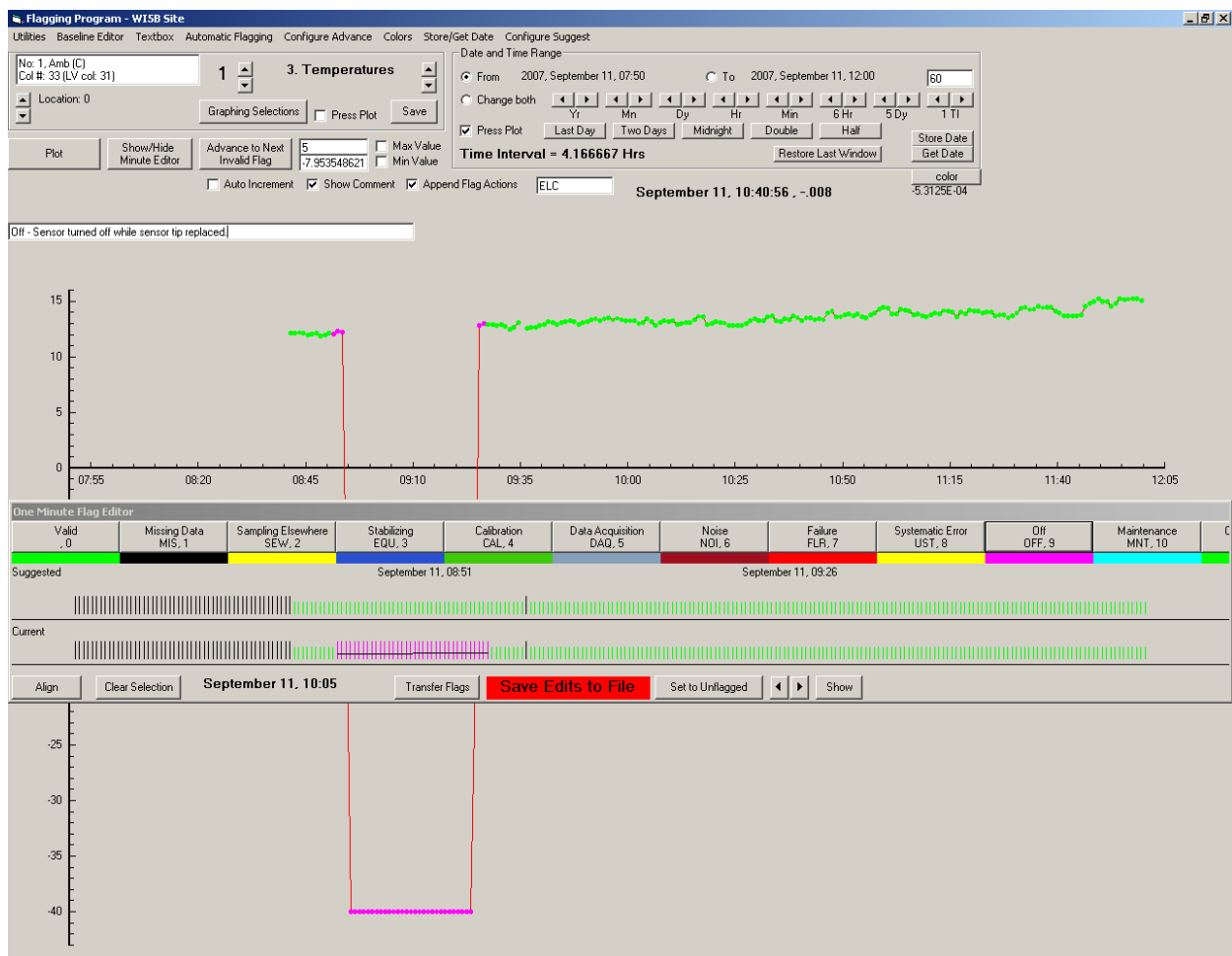


Figure 6. An example of manually flagging data using the One Minute Flag Editor.

- 6.4. Perform calculations and import data using ImportExport program.
- 6.4.1. Open the program; Click “Go To” on the menu bar and select the “Calculate” function.

- 6.4.1.1. Select the file that has the equations to be used for data processing (Fig. 7).
- 6.4.1.2. Select the worksheet that contains the equations, each file can have multiple equation worksheets, for different measurement data analysis.

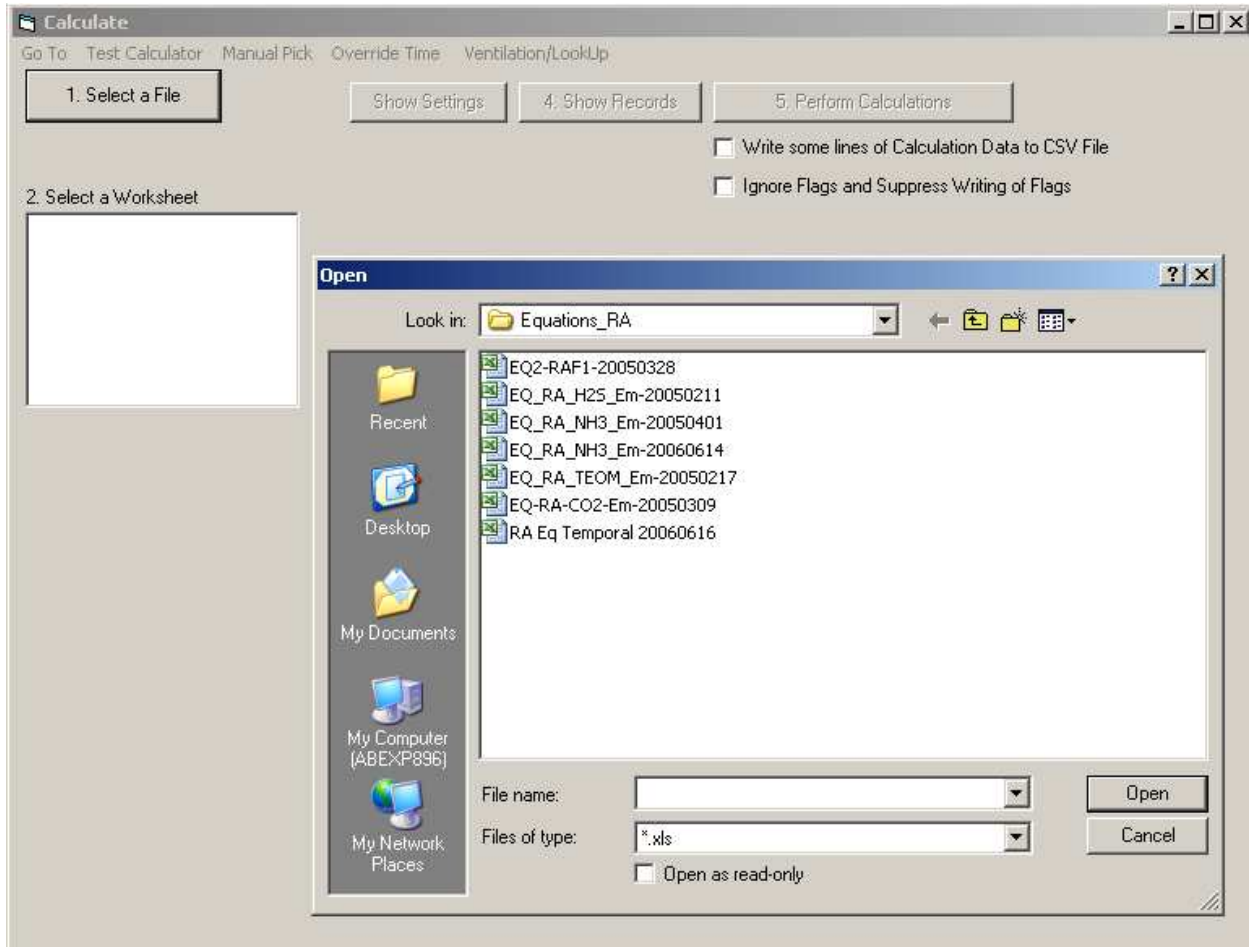


Figure 7. An example of ImportExport program, showing the screenshot of selecting the equation files to execute calculation.

- 6.4.1.3. An equation similar to the following will be used to adjust gas concentration measurement based on precision check data:

$$3/6/06\ 13:55; 3/8/06\ 13:34; c172=c170*A1; A1s =0.97; A1e=0.99$$

where the two sets of date and time specify the calculation period; c172 is the adjusted gas concentrations; c170 is the gas measurement data; and A1 is the linearly interpolated values based on the precision checks: A1s is the beginning precision check ratio (span/as-found ratio), and A1e is the ending precision check ratio.

- 6.4.1.4. An equation similar to the following will be used to calculate emission rate for a specific air stream:

$$c223 = \text{EmissionV}(c18, 17.03, c41, c143, \text{valid}(c180, \text{ave1}(c177, c181)), c183, 1000)$$

where c223 is the calculated ammonia (NH₃) emission rate and EmissionV is a custom function that utilizes many required variables, including atmospheric pressure (c18), molecular weight of NH₃ (17.03), exhaust air stream temperature (c41), airflow rate (c143), exhaust or neighboring exhaust concentrations (c189, c177 and c181), incoming airflow (background) concentration, and a multiplication factor of 1000. The valid function selects the value within the parenthesis when the first column (c180) of data is missing or invalid. The ave1 function return the mean value of c177 and c181, and returns the other value when either one is missing or invalid).

- 6.4.2. ImportExport is also used to import non-AirDac data from various sources (e.g., manure properties data).
- 6.4.2.1. Prepare a comma-separated data file from a template file. Do not change the format of the cells in the first 5 rows and 4 columns. However, change the CAPECAB database column number to indicate the destination of the data.
 - 6.4.2.2. Copy and paste the date and time, and data values into the corresponding cells. The duration between each date and time values is flexible, and the values between these two times can be based on a step change, linearly interpolated, or no change.
 - 6.4.2.3. Save and exit the comma separated file before importing the data.
 - 6.4.2.4. Start the ImportExport program, and select the “Misc Import” function.
 - 6.4.2.5. Click on “Pick a file” to select the data file for the program to read data.
 - 6.4.2.6. Click on “Get Transfer Array” to check the details of the format and data to be imported.
 - 6.4.2.7. Click on “Process the File” to import the data.
- 6.4.3. ImportExport can also be used to import non-AirDac date using pre-configured import settings. This function is generally reserved for animal data collected from the producer (e.g. animal inventory, feed consumption).
- 6.4.3.1. Prepare an ImEx_Info6_XX.csv file, using a previous file as a template.
 - 6.4.3.2. Fill in the required information pertaining to the source columns of data in an Excel spreadsheet, and the destination columns of the CAPECAB database.
- 6.5. Check and export data using the Display program.
- 6.5.1. The program consists of two major functions: ShowGraph and ShowTextBox. They share many features. Use ShowGraph to display results as graphs, and ShowTextBox to display data in tabular format.
 - 6.5.2. An important feature of the Display program is allowing a user-defined data completeness percentage such as 75% when calculating period means to avoid bias. Only mean values which meet or exceed the user-defined data completeness percentage for valid data are presented. This is typically 75%, but will be specified in the QAPP
 - 6.5.3. ShowGraph allows plotting one to four columns of data (Fig. 8).

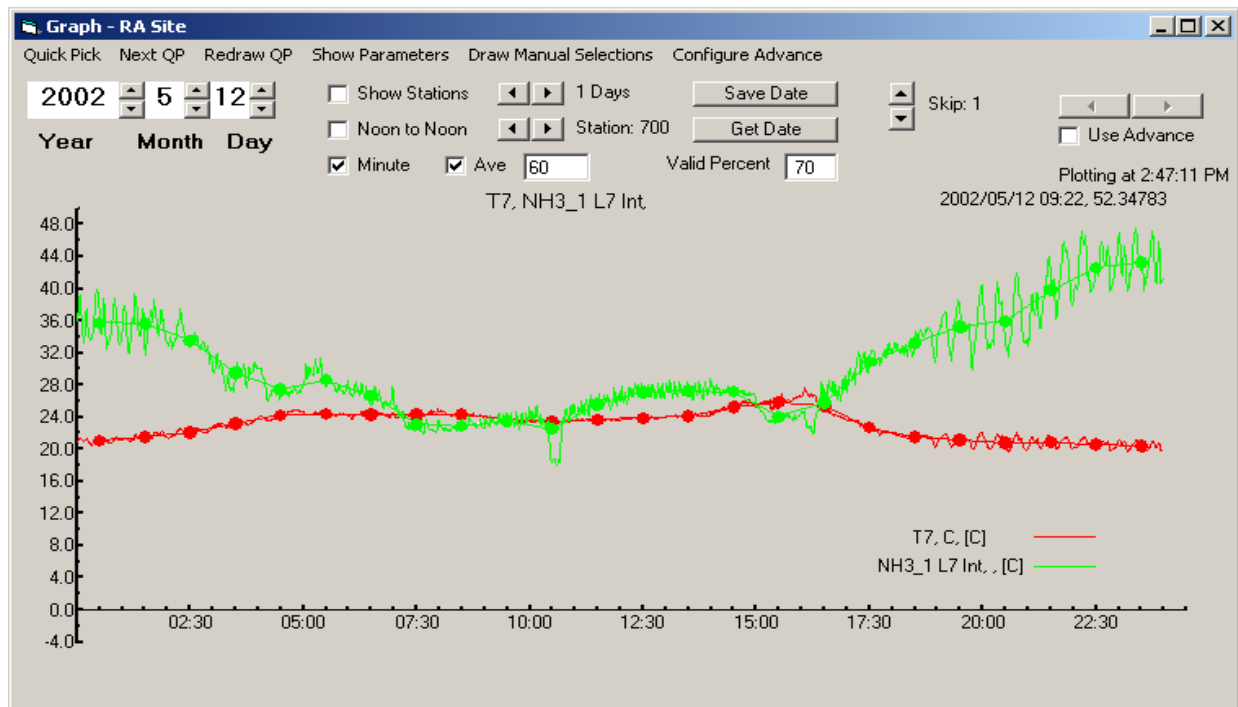


Figure 8. An example of Display-ShowGraph program, displaying the correlation between ammonia concentration and exhaust temperature.

- 6.5.3.1. The program displays one-minute averages and/or any user-specified averages. For example, to check and export three-hour averages of data, set the number of minutes to read to 180. Fig. 8 shows temperatures and NH₃ concentrations of sampling location 7; in this figure, the thin lines are one-minute averages, while the dots indicate 60-min averages.
- 6.5.3.2. When viewing smaller samples of data, the individual points are shown in colors that indicate valid or invalid flags.
- 6.5.4. ShowTextBox presents and exports any number (or all) of data columns.
- 6.5.4.1. Users can see and export unlimited number of rows of data (Fig. 9).
- 6.5.4.2. The program also allows users to define average periods (Fig. 10). Other useful functions include selecting the minimum percent data completeness for mean calculations, and showing blank when there is not enough valid data to meet the required completeness level.

7. Records Management

- 7.1. Manage data files according to SOP B5.
- 7.2. Whenever data is flagged as invalid in the flagging or data-inspection process, record the reason and period in the data processing notebook (or electronic file).
- 7.3. Record other information including date, analyst name, site name, and narrative.

2001 December 01, 00:00 Number of Minutes (Hours): 14400 (240)

July 05, 2006 14:52:38

Date/Time,	T SF17,	NH3_1	L7 I nt
2001/12/01 00:00:00,	17.151,	50.616	
2001/12/02 00:00:00,	17.802,	54.375	
2001/12/03 00:00:00,	19.122,	51.472	
2001/12/04 00:00:00,	20.489,	37.667	
2001/12/05 00:00:00,	21.302,	28.816	
2001/12/06 00:00:00,	17.837,	36.31	
2001/12/07 00:00:00,	16.988,	37.969	
2001/12/08 00:00:00,	15.989,	36.192	
2001/12/09 00:00:00,	15.264,	42.644	
2001/12/10 00:00:00,	15.765,	47.536	

Figure 9. An example of Display-ShowText window, displaying daily mean ammonia concentrations and exhaust temperatures for 240 h (10 d).

2001 December 01, 00:00 Number of Minutes (Hours): 14400 (240)

Date and Number of Records

Yr Mn Dy Hr Min Number of Minutes Skip Bold

Last Quarter Day Station: 700 Get Date Save Date

Minute Averages 1440 Fontsize: 8

Show Flags Interpolate Values 130 Valid Percent 70

Show Blank for Invalid data Force times to beginning of period

Show number of valid data instead of average

Choose or create your own date format

yyyy/mm/dd hh:nn:ss Export with Unformatted DateTime Column

Example: 2003/09/27 15:37:51

Figure 10. An example of Display-ShowText setting window, displaying the selections for displaying data shown in Fig. 8.

8. Quality Control and Quality Assurance

- 8.1. Compare data converted into the CAPECAB database to the original tabular data, for any inconsistencies. Check at least several days of data to make sure the one-minute values agree, and data columns are consistent.
- 8.2. If the format of tabular data is changed (e.g., removed, added, or switched columns), make sure the changes and time are recorded in the data processing notebook and update the Convert files accordingly.
- 8.3. Check the flagging program and processed data to make sure only equilibrium data is extracted as intended.
- 8.4. Closely monitor the measurement data and calculations, especially the diurnal and seasonal variations. When processing weeks or months of data, spot check results to insure accuracy and correctness. A spot check of one day per three months of data should be conducted. The important variables, such as airflow and emission values should be calculated by another person, and compared with the results obtained using CAPECAB.
- 8.5. If a variable(s) is observed to be irregular or doubtful, select other variables to inspect and verify by using the CAPECAB program. Inspecting the closely correlated variables (such as concentration, temperature and ventilation rate) or measurements at adjacent sampling location(s) is essential for data quality.
- 8.6. Users can easily modify the time period (1 h, 2 d, several weeks) of averages and unlimited number of columns and rows of data for inspection.
- 8.7. Inform the measurement site personnel when an error is found and/or corrective action is needed.

9. References

- 9.1. SOP B2. 2006. Data Acquisition and Control Software (AirDAC). Standard Operating Procedure B2. Purdue Ag Air Quality Lab.
- 9.2. SOP B3. 2006. Air Data Pre-Processing Software. Standard Operating Procedure B3. Purdue Ag Air Quality Lab.
- 9.3. SOP B4. 2006. Calculation and Reporting of Air Emissions from Barns. Standard Operating Procedure B4. Purdue Ag Air Quality Lab.
- 9.4. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 9.5. SOP G9. 2006. Precision Checks of Gas Analyzers. Standard Operating Procedure G9. Purdue Ag Air Quality Lab.

10. Contact Information

- 10.1. PAAQL: odor@purdue.edu, heber@purdue.edu, <http://www.AgAirQuality.com>

**OPERATION OF THE THERMO ELECTRON MODEL
146C MULTI-GAS DILUTER
Standard Operating Procedure (SOP) C3**

**OPERATION OF THE THERMO ELECTRON MODEL
146C MULTI-GAS DILUTER
Standard Operating Procedure (SOP) C3**

**Prepared by
Congna Li**

**Reviewed by
Bill W. Bogan and Richard H. Grant**

Effective Date: November 6, 2006

**PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)
Agricultural and Biological Engineering, Purdue University
West Lafayette, IN 47907**

Table of Contents

	Page
1. Scope and Applicability.....	3
2. Summary of Method.....	3
3. Definitions.....	4
4. Health and Safety.....	4
5. Cautions.....	4
6. Interferences.....	4
7. Personnel Qualifications	4
8. Equipment and Supplies	5
9. Procedure.....	5
10. Data and Records Management	13
11. Quality Control and Quality Assurance.....	13
12. References.....	14

1. Scope and Applicability

- 1.1. Precise dilution of manufacturer-supplied gases is required during the calibration of the open-path monitoring instruments that are used to monitor emissions from confined animal feeding operations (such as corrals) and open sources (such as lagoons) at livestock facilities.
 - 1.1.1. Typical dilution ratios needed to calibrate open-source monitoring instruments, such as UV-DOAS (SOP C1) and TDLAS (SOP C2) systems are approximately 100:1 to 1000:1.
- 1.2. This procedure describes the setup and operation of the Thermo Electron Corporation Model 146C Dynamic Gas Calibrator.
 - 1.2.1. The Model 146C can handle zero air flows of 0.2-10 L/min, and span gas flows of 1.0-50 sccm.
 - 1.2.2. The accuracy of each mass flow measurement is 2% of reading or 1% of full scale, whichever is less.
 - 1.2.3. Linearity of mass flow measurement is 0.5% of full scale.
 - 1.2.4. Repeatability of mass flow measurement is 2% of reading or 1% of full scale, whichever is less.
 - 1.2.5. Permeation oven temperature stability is 0.1°C.

2. Summary of Method

The Thermo Environmental Model 146C Multigas Calibrator precisely dilutes calibration gases, which can then be used for precision and Level I span checks, audits, and multipoint calibration of various instrumentation. The Model 146C integrates mass flow controllers, an ozone generator, a permeation tube oven, power supplies, and solenoid valves into a single microprocessor-controlled unit, which can also be controlled remotely using a datalogger or computer. The calibrator uses either permeation tubes (optional add-on) or standard gases as calibration gas sources. The standard Model 146C hardware/plumbing configuration mixes gas and zero air flows. The basic unit can handle three gas standards, which are controlled by individual solenoids and regulated by mass flow controllers; the zero air controller is high-flow, while the gas flow controller is low-flow. The desired concentration is produced in a Teflon mixing chamber. When a permeation gas source is used in conjunction with the gas dilution system, permeation dilution concentration is calculated based on the permeation rate and the flow rate of zero air controlled by the gas dilution system. The permeation oven heats a permeation tube that supplies an internal source of calibration gas. A small amount of zero-air flows through the permeation tube oven and is mixed with the remainder of the zero-air flow in the mixing chamber. Accuracy is achieved through precise control of the release rate of the permeation tube gas and the flow of zero air through the zero-air mass flow controller, as well as maintenance of a stable permeation tube temperature. When only the permeation oven is activated, up to five gas concentration levels (ppm) can be set.

3. Definitions

- 3.1. LED Light-emitting diode
- 3.2. MFC Mass flow controller
- 3.3. NIST National Institute of Standards and Technology
- 3.4. QAPP Quality Assurance Project Plan
- 3.5. sccm Standard cubic centimeters per minute
- 3.6. TDLAS Tunable diode laser acoustic spectroscopy
- 3.7. UV-DOAS Ultraviolet differential optical absorption spectroscopy

4. Health and Safety

- 4.1. Be careful when connecting the precision standard and dilution gas cylinders to the diluter, especially in the case of toxic, reactive, or otherwise hazardous gases. See SOP G2 for more information on handling gas cylinders.
- 4.2. Avoid mixing gases that may produce an explosion or other hazardous reaction.
- 4.3. Be careful when making electrical connections, disconnecting and connecting connectors and making voltage measurements in the instrument as high voltage or current can injure or kill.
- 4.4. Disconnect power before servicing the unit.

5. Cautions

- 5.1. Verify that the scheduled calibration date is prior to the expiration date specified on the EPA protocol calibration gas cylinder. Do not use expired cylinders.
- 5.2. Leave the power on at all times when a permeation tube has been installed. The zero air-air supply should also be connected and active to allow a small flush flow across the permeation tube.
- 5.3. When determining the actual flow through the mass flow controller, use the LED reading, and not the value set on the potentiometer.

6. Interferences

- 6.1. Reactive compounds can be lost in this instrument. Follow the procedure to verify that reactive compounds are not lost in the gas dilution system, especially in Methods 15*, 16, and, 25A and 25B (40 CFR part 60, appendix A) when using a gas other than propane.

7. Personnel Qualifications

- 7.1. Each operator must read and understand this SOP and SOP G2 before working with the instrument. Any operator using this instrument to calibrate the UV-DOAS and/or TDLAS instruments must also read and understand SOP C1 and/or C2, respectively.

- 7.2. Each operator must be trained in the use of the handheld multimeter, the barometer, water bath, and mass balance before initiating the procedure.

8. Equipment and Supplies

- 8.1. Model 146C Dynamic Gas Calibrator (Thermo Electron Corporation, Waltham, MA)
- 8.2. Permeation tube oven and permeation tube(s) containing appropriate gas(es) (optional)
- 8.3. Internal ozone generator (optional)
- 8.4. Teflon tubing
- 8.5. Zero air and span gas cylinders (EPA protocol standard, 1% certification)
- 8.6. Stainless steel dual-stage gas cylinder regulators as appropriate for the particular gas(es) (SOP G2)
- 8.7. Wrench to manipulate gas cylinder connections
- 8.8. NIST-traceable flow calibrator
- 8.9. NIST-traceable thermometer with a range of 0-45°C
- 8.10. Water bath (heatable to 45°C)
- 8.11. Barometric pressure sensor
- 8.12. Balance with 0.01 mg accuracy
- 8.13. Ambient air temperature sensor
- 8.14. 4.369-K Ω Resistor or resistance box capable of providing 4.369 K Ω
- 8.15. Wire leads with clips to connect to resistance box
- 8.16. Multimeter

9. Procedure

- 9.1. Instrument Setup
 - 9.1.1. Connect a source of zero air to the inlet port labeled "ZERO AIR" (Fig. 1).
 - 9.1.2. The zero air source must be capable of supplying the full-scale flow required by the zero air controller (up to 10 L/min) at a pressure between 10 and 40 psi.
 - 9.1.3. Connect the standard gas cylinders to the ports labeled A, B and C (Fig. 1).

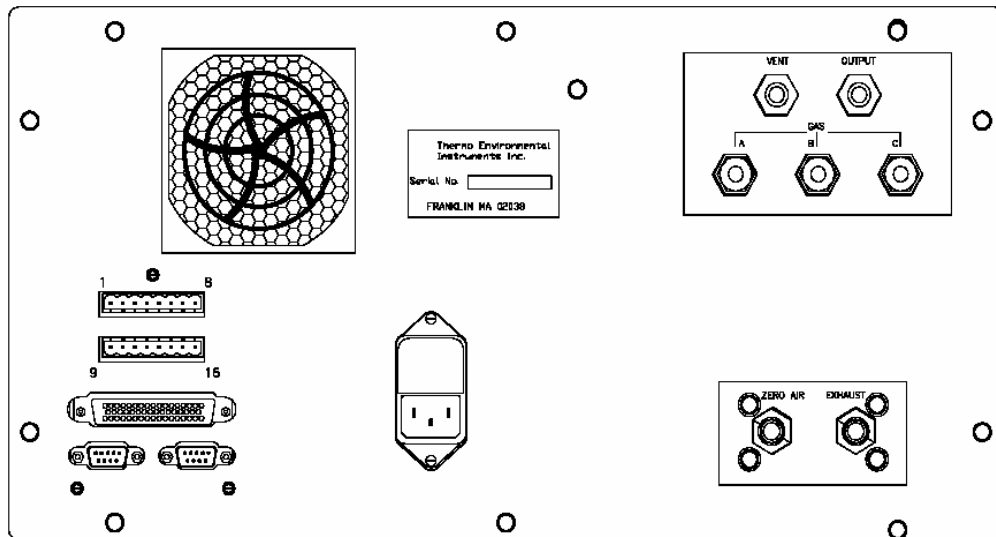


Figure 1. Model 146C Rear Panel.

9.1.4. Install the permeation tube using the following procedure (applicable only if the permeation option is installed):

9.1.4.1. Remove the instrument cover and locate the permeation oven (Fig. 2).

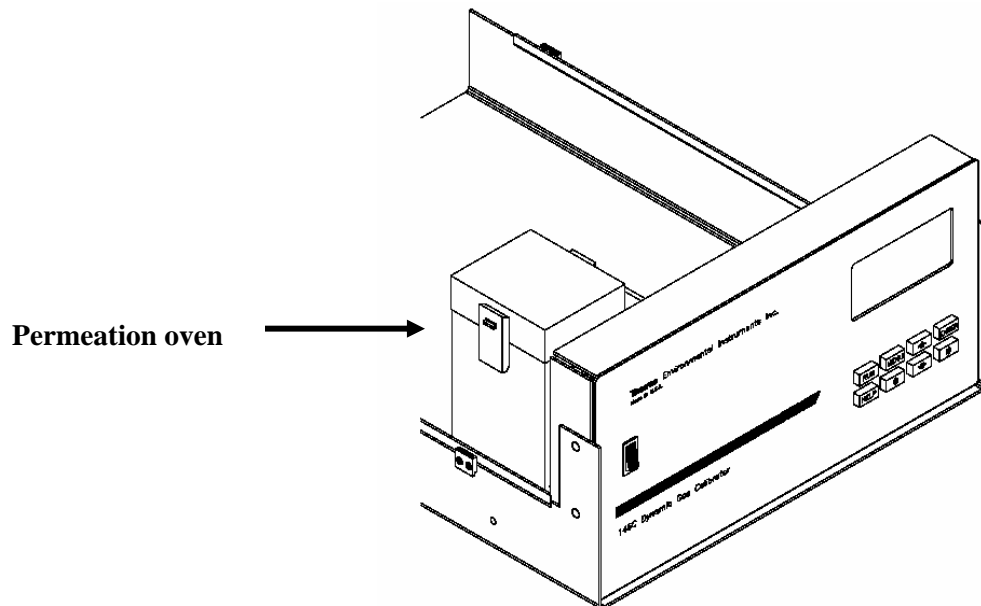


Figure 2. Removing cover of the TEI 146C to access the permeation oven.

- 9.1.4.2. Release the latches on the sides of the oven cover, and remove the cover.
- 9.1.4.3. Remove glass chamber assembly by loosening (not removing) knurled screw, located at the top of the chamber, and gently pulling assembly upward. Completely remove assembly from oven (Fig. 3).
- 9.1.4.4. Separate the glass chamber from the top assembly by twisting and gently pulling glass away from top. Keep glass clean by using Kimwipes or similar material to handle glass.
- 9.1.4.5. Place permeation tube in chamber.
- 9.1.4.6. Attach glass chamber to top assembly by gently pushing together with a slight twisting motion.
- 9.1.4.7. Replace glass chamber assembly into oven until top of assembly is flush or slightly below oven top (Fig. 4).
- 9.1.4.8. Tighten knurled screw with finger. Do not use tools to tighten.
- 9.1.4.9. Replace oven cover, being careful to place tubing and wire in slot of cover.

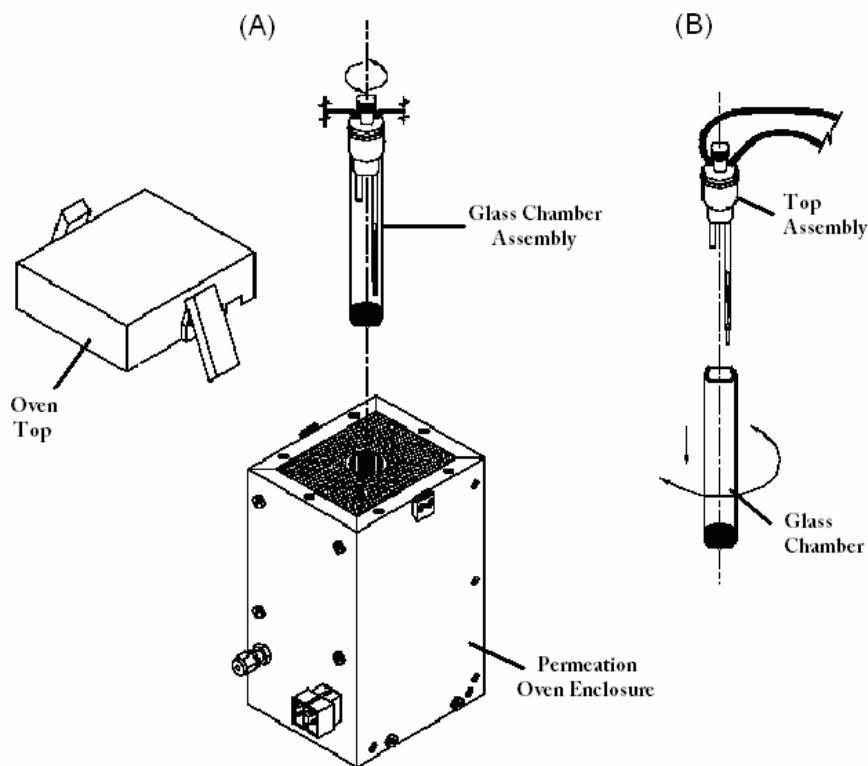


Figure 3. Removing glass chamber from permeation oven (A) and removing top assembly (B).

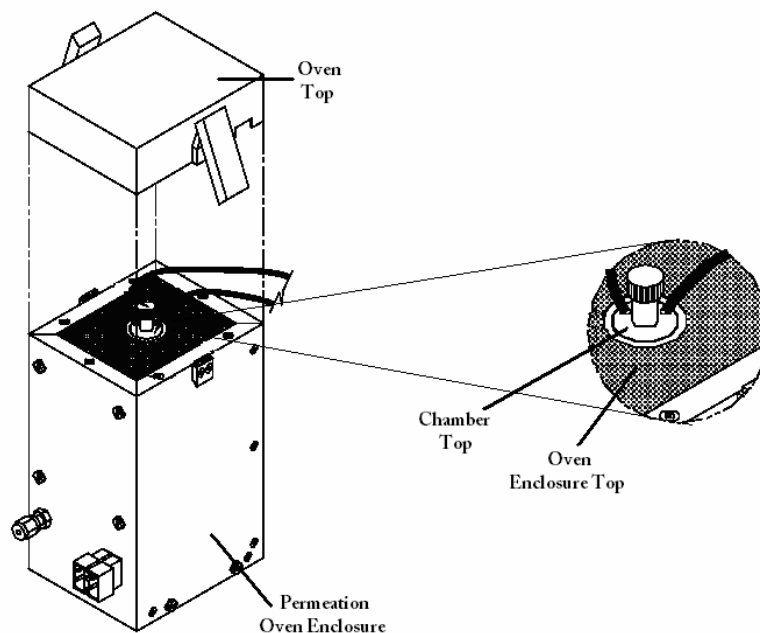


Figure 4. Replacing glass chamber. Inset shows chamber top flush with oven enclosure top.

9.2. Operation

9.2.1. Gas dilution using a gas cylinder

9.2.1.1. Press the “RUN” pushbutton, choose “Run Screen 1”, press the “MENU” pushbutton, select the “Local” mode using the \uparrow and \downarrow pushbuttons, and press “ENTER”. Press “MENU” to return to the Main menu.

9.2.1.2. From the “Main Menu”, move to Gas A, B, or C by using the \uparrow and \downarrow pushbuttons. Press “ENTER” to select.

9.2.1.3. Select “Name”, move the underscore to select a character, using the \leftarrow and \rightarrow pushbuttons. Select a new character using the \uparrow and \downarrow pushbuttons. Press the “ENTER” pushbutton to save the selection.

9.2.1.4. Select “Tank Conc”, enter the gas concentration using the \leftarrow and \rightarrow and the \uparrow and \downarrow pushbuttons. Press “ENTER” to save the selection.

9.2.1.5. Select “Span 0 Flow”, enter the value of the zero-air flow using the \leftarrow and \rightarrow and the \uparrow and \downarrow pushbuttons. Press “ENTER” to save the selection.

9.2.1.6. Use the \leftarrow and \rightarrow pushbuttons to select the desired span level (Span 1 to 5, or manual).

9.2.1.7. Select “Span”, select the span gas concentration and total flow rate (sum of zero-air and span gas flow) using the \uparrow and \downarrow pushbuttons. Press “ENTER” to switch to the corresponding screen.

- 9.2.1.8. Under the “Span” menu, select “Conc PPM”, enter the value of the diluted gas concentration using the ← and → and the ↑ and ↓ pushbuttons. Press “ENTER” to save your selection.
- 9.2.1.9. Under the “Span” menu, select “Span Flow”, enter the value of the span gas flow rate using the ← and → and the ↑ and ↓ pushbuttons. Press “ENTER” to save your selection.
- 9.2.1.10. To manually set the zero-air and gas flows, select “Manual” after selecting Gas A, B, or C, use the ↑ and ↓ pushbuttons to select zero-air and gas flow. Press “ENTER”. Then select “ZERO AIR SCCM” to display and modify the zero air flow setting manually, or select “Gas SCCM” to display and modify the span gas flow setting manually using the ← and → and the ↑ and ↓ pushbuttons. Press “MENU” to return to the upper menu or press “ALT” to return to the “Run” screen.
- 9.2.1.11. Computation of diluted gas concentrations
- 9.2.1.11.1.

$$\text{Model 146C output (ppm)} = \frac{F_{\text{gas}}}{F_{\text{gas}} + F_{\text{zero air}}} \times C_{\text{cyl}}$$

Where:

- F_{gas} = Flow measured for cylinder gas, sccm.
 $F_{\text{zero air}}$ = Flow measured for zero air, sccm.
 C_{cyl} = Concentration of gas in cylinder, ppm

9.2.2. Gas dilution using the permeation tube oven

- 9.2.2.1. Allow 24 h for permeation tube temperature to stabilize after turning the unit on. If a permeation tube is purchased with a certified rate, R2, at a given temperature, T2, it is possible to calculate the approximate temperature T1 that is needed to achieve permeation rate R1, by using the following equation:
- 9.2.2.1.1.
- 9.2.2.1.2. $\text{Log } R_1 = \text{log} R_2 - 2950(1/T_1 - 1/T_2)$

Where:

- R_1 = Permeation rate in ng/min at T_1 ,
 R_2 = Permeation rate in ng/min at T_2 .

- 9.2.2.2. From Run Screen 1, use the ↑ and ↓ pushbuttons to scroll to the ozone/perm line.
- 9.2.2.3. Use the ← and → pushbuttons to select the desired permeation level (Perm Levels 1 through 5). Press “ENTER”.
- 9.2.2.4. Set the flow of zero air in the manual settings screen of the flow controls menu.
- 9.2.2.5. Allow 24 h for the permeation tube temperature to stabilize.
- 9.2.2.6. Calculate the permeation rate, R, following Section 9.3.2.3.
- 9.2.2.7. Computation of diluted permeation gas concentrations

9.2.2.7.1. Model 146C output (ppm) = RK/Q_o

Where:

R = Permeation rate, ng/min

Q_o = Zero air flow rate, sccm

K = Constant for specific permeant = 24.45/ MW

MW = Molecular weight of specific permeant

9.3. Calibration

9.3.1. Calibration of mass flow controllers

9.3.1.1. Connect a source of clean, dry air to the inlet of the mass flow controller.

9.3.1.2. Measure barometric pressure and room temperature.

9.3.1.3. Connect a suitable flow meter (NIST-traceable calibrator) to the mass flow controller outlet.

9.3.1.4. Set the Model 146C to “Gas Drive” or “Zero Air Flow Calibration”.

9.3.1.5. Enter the flow meter reading using the flow input screen.

9.3.1.6. Repeat the above two steps for the remaining flow settings.

9.3.1.7. If difficulty is encountered due to a malfunction of the flow controller, contact Thermo Environmental Instruments.

9.3.2. Calibration of permeation tube oven

9.3.2.1. Setting water bath to the desired temperature

9.3.2.1.1. Unplug the connector at J3, from the Oven Controller Board (Fig. 5). Place a 4.369-K Ω resistor across pins 3 and 4 of J3 on the Oven Controller Board.

9.3.2.1.2. From the Permeation Oven Menu, select Cal Oven Thermistor. Adjust R2 on the Oven Controller Board until the oven drive voltage is 5.000 volts. Press MENU to return to the Permeation Oven Menu.

9.3.2.1.3. Remove the thermistor from the permeation tube oven. Leave the thermistor connected to the Oven Controller Board. Insert the thermistor into a water bath next to the NIST-traceable thermometer.

9.3.2.1.4. Turn on the power to the water bath. Using a NIST-traceable thermometer with a resolution of $\pm 0.01^\circ\text{C}$, adjust water bath to $45.00 \pm 0.02^\circ\text{C}$.

9.3.2.1.5. From the Permeation Oven Menu, select Cal Gas Thermistor. Adjust R4 on the Oven Controller Board until the permeation gas temperature reading is 45.00°C .

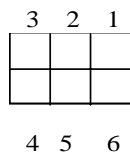
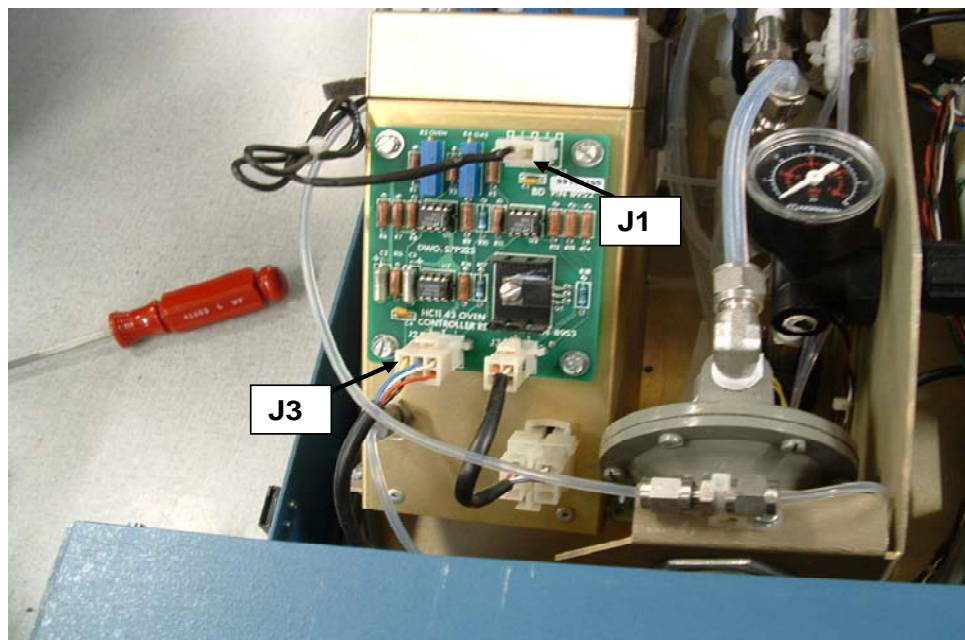
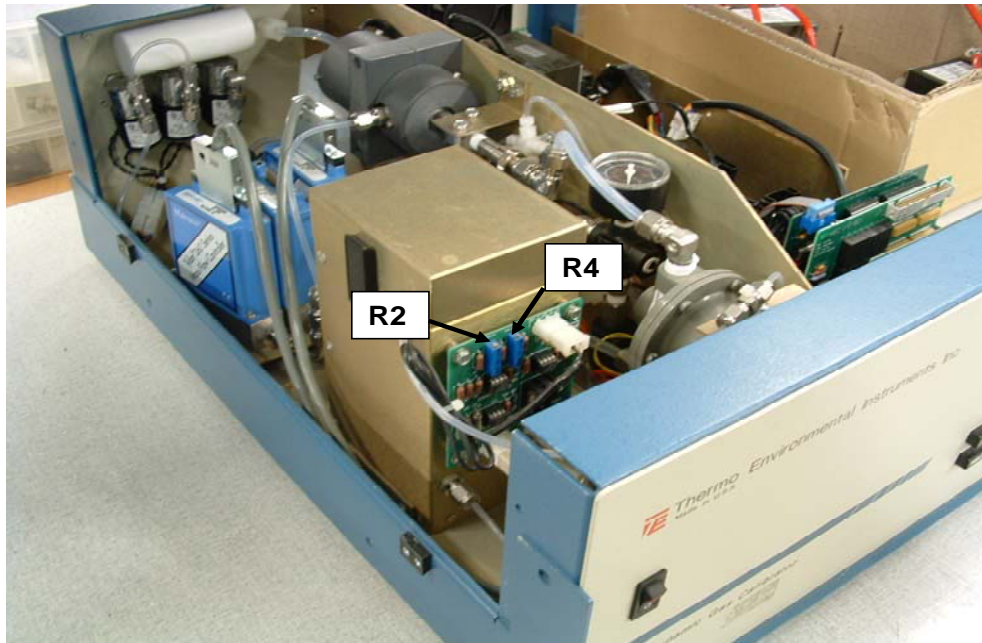
9.3.2.1.6. Remove thermistor from the water bath, dry, and replace into the permeation tube oven.

9.3.2.1.7. Wait for the permeation gas temperature reading to stabilize.

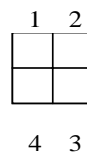
9.3.2.1.8. From the Permeation Oven Menu, select Set Gas Temperature. Adjust R2 until the Perm Gas reading displayed on the first line is 45.00°C . Wait at least 10 min between adjustments.

9.3.2.2. Setting measure temperature with accurately known oven temperature (as an alternative approach to that described in Section 9.3.2.1)

9.3.2.2.1. Unplug the connector at J3, from the Oven Controller Board. Place a 4.369 K Ω resistor across pins 3 and 4 of J3 on the Oven Controller Board.



J3 Pin Layout



J1 Pin Layout

Figure 5. The permeation oven controller board, showing the board's location, the locations of R2, R4, J1 and J3 on the board, and the pin layouts of J1 and J3.

- 9.3.2.2.2. From the Permeation Oven menu, select Cal Oven Thermistor. Adjust R2 on the Oven Controller Board until the oven drive voltage is 5.000 volts. Press MENU to return to the Permeation Oven menu.
- 9.3.2.2.3. Unplug the thermistor from J1 on the Oven Controller Board.
- 9.3.2.2.4. Connect a resistance of 4.369 K Ω across pins 1 and 2 of J1.
- 9.3.2.2.5. From the Permeation Oven menu, select Cal Gas Thermistor. Adjust R4 on the Oven Controller Board until the permeation gas temperature reading is 45°C.
- 9.3.2.2.6. Reconnect the thermistor to J1.
- 9.3.2.2.7. Wait for the permeation gas temperature reading to stabilize.
- 9.3.2.2.8. From the permeation oven menu, select Set Gas Temperature. Adjust R2 until the Perm Gas reading displayed on the first line is 45.00°C. Since it takes several minutes for the permeation oven temperature to stabilize, wait 10 min between adjustments.
- 9.3.2.3. Determination of permeation rate by weight loss
 - 9.3.2.3.1. Make sure the oven has been calibrated as described above.
 - 9.3.2.3.2. Gently insert the permeation tube using clean tweezers or similar tool. Never touch the tube with your fingers.
 - 9.3.2.3.3. Turn on the Model 146C.
 - 9.3.2.3.4. Wait 48 to 72 hours for the permeation tube temperature to stabilize.
 - 9.3.2.3.5. Carefully remove the permeation tube from the oven and weigh permeation tube to nearest 1 mg. Perform this measurement as quickly as possible.
 - 9.3.2.3.6. Return permeation tube to the oven.
 - 9.3.2.3.7. Allow permeation tube to permeate at constant temperature with gas flow over tube for a period sufficient to generate a measurable weight loss (usually a minimum of 72 h).
 - 9.3.2.3.8. Reweigh permeation tube to nearest 1 mg.
 - 9.3.2.3.9. Compute the permeation rate, R, in terms of ng/min as:
$$R = (\text{Initial weight} - \text{Final weight}) / \text{Time.}$$
 - 9.3.2.3.10. Use the permeation tube in the same oven that was used to determine the permeation tube's weight loss.
- 9.3.2.4. Determination of release rate by use of transfer standard
 - 9.3.2.4.1. To perform this procedure, one Model 43C and two Model 146Cs are needed.
 - 9.3.2.4.2. Ensure that the ovens in both of the Model 146Cs have been calibrated.
 - 9.3.2.4.3. Determine permeation rate for permeation tube in Model 146C being used as calibration standard, or install a certified permeation tube.
 - 9.3.2.4.4. Allow the permeation tubes in both Model 146Cs to stabilize at least 48 h.
 - 9.3.2.4.5. Carefully calibrate an analyzer, such as a Thermo Environmental Model 43C, using the Model 146C with the calibrated permeation tube. Follow the instruction for calibration in the 43C instrument manual.
 - 9.3.2.4.6. Connect calibrated analyzer to Model 146C with permeation tube whose permeation rate is to be determined.

- 9.3.2.4.7. Adjust flow of the Model 146C so that the analyzer reads close to full scale. Note flow and measured concentration.
- 9.3.2.4.8. Compute permeant concentration from the following equation:

$$\text{Model 146C output (ppm)} = \frac{R \times K}{Q_o}$$

Where:

R = Permeation rate, ng/min

Q_o = Flow rate, sccm

K = Constant for specific permeant = $\frac{24.45}{MW}$

MW = Molecular weight of specific permeant

10. Data and Records Management

- 10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this instrument. Supplement this electronic record by a record book designated for this instrument, which should contain copies of the electronic record.
- 10.2. Manage all data according to SOP B5.
- 10.3. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes. Overstrike all errors in writing with a single line. Initial and date all such corrections.
- 10.4. A copy of the manufacturer' instructions for the operation of the instrument, as well as the most recent calibration documentation shall be made available for the project auditor's inspection upon request.
- 10.5. A label (Fig. 6) shall be affixed at all times to the gas dilution system listing the date of the most recent calibration, the due date for the next calibration, and the person or manufacturer who carried out the calibration.

Calibration date: _____ Next calibration due date: _____ Calibrated by: _____

Figure 6. Sample calibration label.

11. Quality Control and Quality Assurance

- 11.1. Check and recalibrate the mass flow controller, using a NIST-traceable flow calibrator, whenever the target and actual flow (within the range of 20 to 100% of full scale) deviates greater than $\pm 2\%$ of reading or 1% of full scale, whichever is less. The QAPP for the particular project may have additional requirements.
- 11.2. Calibrate the permeation tube oven whenever the temperature indicator deviates by 0.2°C (an error of about 0.1°C corresponds to an error of 1% in release rate).

- 11.3. Calibrate the flow calibrator, thermometer, water bath, barometric pressure sensor, balance and ambient air temperature sensor at the initiation of the study and annually thereafter, unless the QAPP specifies otherwise.

12. References

- 12.1. Thermo Electron Corporation. 2004. Model 146C Dynamic Gas Calibrator, Operating Manual.
- 12.2. U.S. Code of Federal Regulations, US 40 CFR 51 Appendix M, Method 205— Verification of Gas Dilution Systems for Field Instrument Calibrations. Available at: <http://ecfr.gpoaccess.gov/cgi/t/text/text-idx?c=ecfr&sid=47660cf35a767ae7d9a8284226be43a7&rgn=div9&view=text&node=40:2.0.1.1.2.20.11.20.24&idno=40>. Accessed 10 January 2006.
- 12.3. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 12.4. SOP C1. 2006. Open Path Measurement of Ammonia and Hydrogen Sulfide with the Cerex UV Sentry Ultraviolet Differential Optical Absorption Spectrometer. Standard Operating Procedure C1. Purdue Applied Meteorology Lab.
- 12.5. SOP C2. 2006. Measurement of Ammonia with the Boreal Laser GasFinder Tunable Diode Laser Absorption Spectrometer (TDLAS). Standard Operating Procedure C2. Purdue Applied Meteorology Lab.
- 12.6. SOP G2. 2006. Standard Operating Procedure for Compressed Gas Cylinders G2. Purdue Ag Air Quality Lab.
- 12.7. SOP G5. 2006. Measurement of Hydrogen Sulfide (H₂S) with the Thermo Electron Corporation Model 45C Pulsed-Fluorescence Analyzer. Standard Operating Procedure G5. Purdue Ag Air Quality Lab.

TEMPERATURE MEASUREMENT USING THERMOCOUPLES
Standard Operating Procedure (SOP) E1

TEMPERATURE MEASUREMENT USING THERMOCOUPLES

Standard Operating Procedure (SOP) E1

Prepared by

Matthew J. Darr (Ohio State Univ.) and Bill W. Bogan

Reviewed by

Sam Hanni, Teng Lim, Albert J. Heber and Kenneth D. Casey (Texas A&M Univ.)

Effective Date: November 6, 2007

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions	3
4. Health and Safety	4
5. Cautions	4
6. Interferences	5
7. Personnel Qualifications.....	5
8. Equipment and Supplies	5
9. Procedures	5
10. Data and Records Management	6
11. Quality Control and Quality Assurance	7
12. References.....	8

1. Scope and Applicability

- 1.1. Temperature measurements at barns during emissions tests are needed for several reasons.
 - 1.1.1. Temperature information is needed to make density corrections in the course of determining pollutant concentrations in air.
 - 1.1.2. Temperature readings are used by the barn's environmental control system to control fans, heaters, and cooling systems.
 - 1.1.3. Temperature is important to assess environmental conditions for instruments, people, and animals.
 - 1.1.4. Temperature is needed to assess and diagnose the environmental processes within the building.
 - 1.1.5. Temperature affects biological activity, which in turn affects gas emissions.
- 1.2. This method is specific for Type T thermocouples, which are used with the National Instruments (NI) Model FP-TC-120 signal input module.
- 1.3. Type T thermocouples are suitable for use within the range of -200 to $+350$ °C, in mildly oxidizing or reducing environments, and in moist air.
- 1.4. Because copper/constantan wires can be manufactured to a greater level of homogeneity than most other thermocouple alloys, the measurement error due to wire heterogeneity is smaller, and Type T thermocouples are more accurate than other types. The overall degree of error associated with this method is $< \pm 2$ °C within the expected temperature range of -30 °C to 40 °C.
 - 1.4.1. The stated standard error for Type T thermocouples is ± 1 °C or $\pm 0.75\%$ of reading (whichever is greater).
 - 1.4.2. Cold-junction accuracy for the NI FP-TC-120 input module is $0.15 - 0.30$ °C.
 - 1.4.3. Linearization algorithms used by the signal input module are accurate to within ± 0.05 °C, and are therefore a negligible source of error.
- 1.5. The locations for positioning thermocouples at a particular site will be specified in the project QAPP, specifically, in the Site Monitoring Plan.

2. Summary of Method

The method described in this SOP measures temperatures through the use of copper/constantan thermocouples (Type T). The thermocouple is the joint formed by the ends of two copper/constantan wires, which have been twisted together. Each thermocouple is individually connected to a signal input module (NI FP-TC-120). This connection is accomplished through the FP-TB-3 isothermal terminal base, such that all terminals are kept at the same temperature. The TC-120 collects data as described in PAAQL SOPs B1 and B2.

3. Definitions

- 3.1. Cold junction The junction formed by joining the thermocouple wires (or their extensions) to the instrument wires. The temperature-dependent voltage created by this junction must be taken into account when determining the temperature at the

- thermocouple tip. This “Cold-junction compensation” function is built into the FP-TC-120 Input Module.
- 3.2. Constantan Any one of a group of copper/nickel alloys that are particularly well-suited for use in thermocouples. Typical constantan compositions used in thermocouples are 40% nickel and 60% copper, with very low thermal expansion coefficients.
- 3.3. DAC Data acquisition and control
- 3.4. Electromotive force (emf) The energy per unit charge that is converted reversibly from (in this case) thermal energy into electrical energy in a metal wire. This quantity will vary depending upon the precise metal composition of the wire or thermocouple.
- 3.5. PAAQL Purdue Agricultural Air Quality Laboratory
- 3.6. SOP Standard Operating Procedure

4. Health and Safety

- 4.1. Make sure that all wiring meets applicable electrical codes.
- 4.2. Extreme care should be taken when installing the wiring near animal cages.

5. Cautions

- 5.1. Thermocouple output signals are prone to picking up electrical “noise”. However, unless thermocouples are run in the direct vicinity of power lines, the electrical fields encountered in barns should not be strong enough to cause enough noise to affect this type of thermocouples. The FP-TC-120 rejects “common mode noise” (noise that is common to both wires in the extension cable); thus, if thermocouples must be run through areas with stronger electrical fields, twist the two wires together to ensure that noise signals will be picked up by both wires. Pre-twisted wire can also be purchased commercially.
- 5.2. Heat dissipated from adjacent equipment (including other thermocouple input modules) can generate thermal gradients by heating up the terminals to different temperatures relative to each other and to the sensor used to measure the cold-junction temperature. The FP-TC-120 is capable of correcting for the errors produced by a gradient of up to 0.2 °C; this is equivalent to a heat dissipation of approximately 4 W. Take steps to ensure that no other equipment in the vicinity of the TC-120 is dissipating more heat than this. Special care should be taken to locate voltage conversion devices (power supplies, computers, AC powered sensors) at least 0.5 meters away from the FP-TC-120 due to their high heat output rates.
- 5.3. Thermocouple wire itself can be a source of thermal gradients, by conducting heat to or from the terminal connection (depending upon the temperature encountered elsewhere by the wire). Minimize this phenomenon taking the following precautions:
- 5.3.1. Run extension wires to different thermocouples together as much as possible to keep all wires at the same temperature.

- 5.3.2. Avoid running thermocouple extension wires near objects that are (or may be) hotter or colder than the rest of the environment.
- 5.4. Store thermocouple wire in a dry, clean location.
- 5.5. Do not use any oils or solvents on any part of a thermocouple assembly.

6. Interferences

- 6.1. Use of other than Type T extension wire will introduce larger error limits, as dissimilar metals or alloys will not display the same emf at a given temperature. Use only Type T thermocouple wire when repairing damaged thermocouples.

7. Personnel Qualifications

- 7.1. Personnel should be trained in the use of the equipment (including thermocouples and their corresponding data acquisition modules) before initiating the procedure.
- 7.2. Personnel should be trained in proper fabrication and maintenance of thermocouples and thermocouple junctions before installation.
- 7.3. Each analyst must read this SOP before initiating the procedure.

8. Equipment and Supplies

- 8.1. Type T (copper/constantan) thermocouple wire, Type T twisted pair, 20 ga (ThermoElectric Wire & Cable, Saddle Brook, NJ), or equivalent
- 8.2. Heat-shrinkable tubing
- 8.3. Heat gun or hand-held torch
- 8.4. Pliers with wire cutters and wire strippers
- 8.5. Temperature probe for field calibration checks. Required specifications are $\pm 0.2^{\circ}\text{C}$ accuracy @ 20°C and NIST traceability.
- 8.6. Hg thermometer with a -10°C to 110°C range (0.5°C scale), checked to agree with the temperature probe to within 1.0°C
- 8.7. Insulated Thermos or similar vessel
- 8.8. Ice
- 8.9. Microwave
- 8.10. FP-TC-120 8-Channel Thermocouple Input Module (National Instruments Corporation, Austin, TX)
- 8.11. FP-TB-3 Isothermal Terminal Base (National Instruments Corporation, Austin, TX)
- 8.12. Soldering iron
- 8.13. Electrical tape

9. Procedures

- 9.1. Fabrication
 - 9.1.1. Remove no more than one inch of insulation from both the copper and constantan wires at the end of the wire that is to serve as the thermocouple.

- 9.1.2. Wrap the copper and constantan wires around each other a minimum of six turns (roughly $\frac{1}{2}$ "). Cut the wires such that a straight cut is made across both (Fig. 1).
- 9.1.3. Protect the junction and exposed wires by using a commercially available heat-shrinkable tubing. Sheathe the entire exposed wire, and $\frac{1}{4}$ "- $\frac{1}{2}$ " of the insulated wire, with the wrap, and cut the wrap approximately $\frac{1}{4}$ " beyond the welded thermocouple junction. Heat with a heat gun or hand-held torch. When the wrap is firmly in place, crimp the end (the $\frac{1}{4}$ " beyond the junction) with pliers, taking care not to crimp over or crush the junction itself (Fig. 1).
- 9.2. Installation
 - 9.2.1. Mount the thermocouple(s) at the specified locations. If airflow is in a prevailing direction, junctions should face into the flow, with the thermocouples themselves trailing in the direction of the flow. Mount thermocouples in locations that are not susceptible to disturbance by animals and/or workers.
 - 9.2.1.1. Mount thermocouples using a flexible coupler or connector. The coupler must allow the thermocouple to bend freely if contacted by animals or facility personnel.
 - 9.2.1.2. In order to ensure thermal insulation from the support structure, ensure that the distance (length of wire) between the attachment point nearest the junction and the junction itself is at least 20 wire diameters.
 - 9.2.2. Prevent excessive sharp bending of the thermocouple wire during installation.
 - 9.2.3. When laying thermocouple wires from the measurement location to the input module, follow all guidelines in Section 5.3, so as to minimize the introduction of thermal gradients to the terminals through the wires, and do not run thermocouple wire near barn power cables, to avoid electrical noise.
- 9.3. Connection to data acquisition module
 - 9.3.1. Configure each channel of the FP-TC-120 for the type of thermocouple to which it will be connected. This process is described in SOP B1.
 - 9.3.2. Connect Type T extension wires from the different thermocouples to the Input Module "IN(+)" and "IN(-)" terminals, as described in SOP B1 (Fig. 2).
 - 9.3.3. On Type T thermocouples, the blue wire (copper) is "+", while the red wire (constantan) is "-".
 - 9.3.4. If shielded thermocouple wires are used, connect one end of the shield to the corresponding "COM" terminal of the module.
 - 9.3.5. Select a temperature range on the FP-TC-120; this is necessary to enable automatic cold-junction compensation. If an input voltage (as opposed to temperature) range is selected, cold-junction compensation will not be performed.
- 9.4. After installation, conduct a two-point check on each thermocouple (Section 11.2.1) to ensure that the thermocouples' performance is within acceptable limits across the entire temperature range that might be encountered in the barn.

10. Data and Records Management

- 10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.

- 10.2. Manage all data according to SOP B5.
- 10.3. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes. Overstrike all errors in writing with a single line. Initial and date all corrections.

11. Quality Control and Quality Assurance

11.1. Initial checks

- 11.1.1. Upon receipt, thoroughly inspect all thermocouple wire and its packing/shipping containers for any sign of damage.

11.2. Calibration

11.2.1. Initial calibration

- 11.2.1.1. After placing them in the barn, check each thermocouple for calibration at temperatures above and below the temperature range encountered in barns.

- 11.2.1.1.1. Insert the thermocouple and a pre-calibrated thermometer into an ice bath in an insulated Thermos or equivalent. Allow temperatures and readings to stabilize for a minimum of 5 min (with stirring or gentle agitation throughout), and record both the thermocouple output and thermometer reading.

- 11.2.1.1.2. In the microwave oven in the instrument trailer (SOP U1), heat enough water to fill the insulated Thermos or similar vessel about $\frac{3}{4}$ full. Check the temperature with the pre-calibrated thermometer to ensure that it is at least 60°C. Immerse the thermocouple and pre-calibrated thermometer in the Thermos, with stirring or agitation, for a minimum of 5 min, and record the thermocouple output and thermometer reading.

- 11.2.1.1.2.1. Re-heat the water when its temperature drops below 50 °C.

- 11.2.1.2. Evaluate the performance of each thermocouple at each temperature according to Section 11.2.3.

11.2.2. Calibration checks during use

- 11.2.2.1. Check each installed thermocouple every six months by comparing it (in place) with the portable NIST-traceable temperature probe (Section 8.5). Assess performance, and take any necessary corrective action, according to Section 11.2.3.

11.2.3. Performance criteria and data corrections for out-of-calibration thermocouples

- 11.2.3.1. If the thermocouple reads $> \pm 2^\circ\text{C}$ different from the reference thermometer, record the magnitude of the difference, replace the thermocouple and re-test.

- 11.2.3.1.1. If the new thermocouple agrees to within 2°C , leave it in place and refer to the following to determine what to do with the old thermocouple and how to treat the data.

- 11.2.3.1.1.1. If the thermocouple, when checked in Step 11.2.2.1, was found to be $\geq \pm 6^\circ\text{C}$ different from the reference thermometer or portable sensor, flag the measurements during the period since the thermocouple was last known to be in calibration (i.e. the previous six months) as questionable.

- 11.2.3.1.1.2. If the thermocouple, when checked in Step 11.2.2.1, was found to be $\geq \pm 4^\circ\text{C}$ different from the reference thermometer or portable sensor, repeat the two-point temperature check (Section 11.2.1) on the thermocouple

before discarding it. Use the regression lines derived from the initial two-point check, and the new one, to correct all temperature data collected since the last calibration, assuming that the drift to the new regression line was linear over time. The amount of correction is therefore time-dependent.

- 11.2.3.1.2. If the reading with the new thermocouple is still $> 2^{\circ}\text{C}$ different from the reference thermometer, and this problem is not observed with other thermocouples in the barn, carefully evaluate the path of the wire for any possible sources of interference, and consider relocating the thermocouple.

12. References

- 12.1. Alltemp Sensors Inc. 2000. Installation-Operation-Maintenance for Thermocouples (T/C). Online at <http://www.wika.ca/wika/techlib/supportfiles/Install/Thermocouples.pdf>. Accessed 1/20/2006.
- 12.2. American Society for Testing and Materials. Test Method E220-02: Standard Test Method for Calibration of Thermocouples by Comparison Techniques.
- 12.3. American Society for Testing and Materials. 1993. Manual on the Use of Thermocouples in Temperature Measurement. ASTM, Philadelphia, PA.
- 12.4. Megatron Electronics and Controls, Ltd.. 1999. Thermocouple Reference Information. Online at <http://www.megatron.co.il/Thermocouple.html>. Accessed 1/20/2006.
- 12.5. Moffat, R.J. 1997. Electronics Cooling: Notes on Using Thermocouples. Online at http://www.electronics-cooling.com/Resources/EC_Articles/JAN97/jan97_01.htm. Accessed 1/20/2006.
- 12.6. National Instruments Corporation. 2002. FP-TC-120 and cFP-TC-120 8-Channel Thermocouple Input Modules: Operating Instructions. National Instruments Corporation, Austin TX.
- 12.7. Pico Technology. Thermocouple Application Note. Online at <http://www.picotech.com/applications/thermocouple.html>. Accessed 1/20/2006.
- 12.8. SOP B1. 2006. Installation, Use and Maintenance of Data Acquisition and Control Hardware. Standard Operating Procedure B1. Purdue Ag Air Quality Lab.
- 12.9. SOP B2. 2006. Data Acquisition and Control Software (AirDAC). Standard Operating Procedure B2. Purdue Ag Air Quality Lab.
- 12.10. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 12.11. SOP U1. 2006. On-farm Instrument Shelters for Barn Sources. Standard Operating Procedure U1. Purdue Ag Air Quality Lab.



Figure 1. Steps in thermocouple fabrication. Shown are the twisted wire pair after cutting (Step 9.1.2) and the fully-attached shrink wrap (Step 9.1.3).

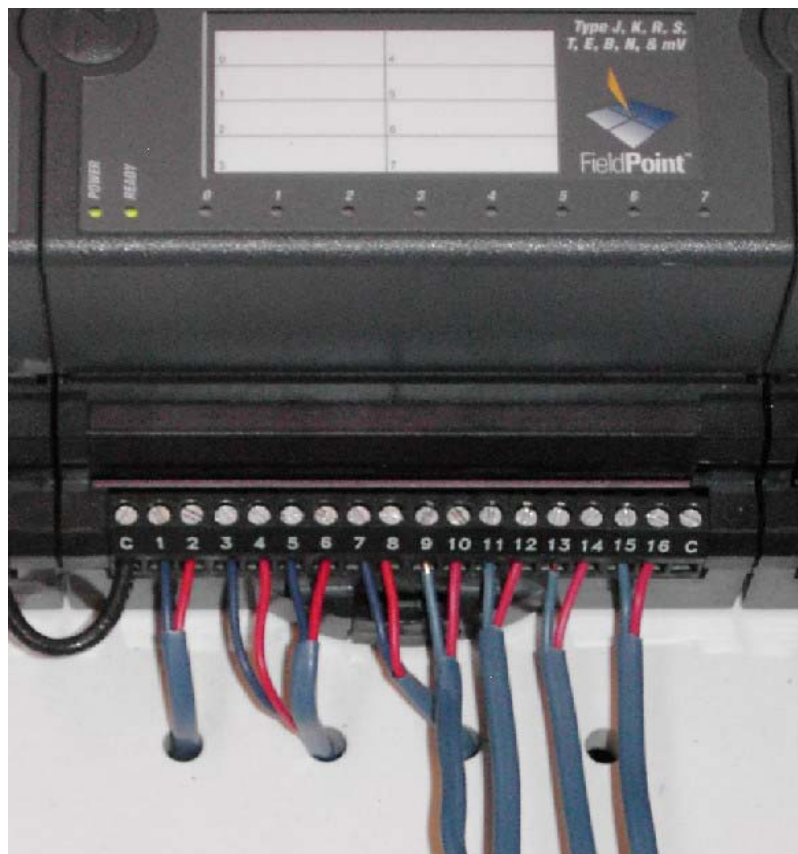


Figure 2. Attachment of thermocouples to the NI FP-TC-120 Thermocouple Signal Input Module. Note connection of blue (copper, +) wires to odd-numbered terminals, and red (constantan, -) wires to even-numbered terminals. Exact wiring scheme is given in SOP B1.

**MANAGEMENT OF OPEN-SOURCE, WEATHER, AND LAGOON-
CHARACTERIZATION DATA
Standard Operating Procedure (SOP) D1**

**Prepared by
Richard H. Grant**

**Reviewed by
Bill W. Bogan, Ji-Qin Ni, and Albert J. Heber**

Effective Date: November 6, 2006

**PURDUE APPLIED METEOROLOGY LABORATORY (PAML)
Agronomy, Purdue University
West Lafayette, IN 47907**

Table of Contents

	Page
1. Scope and Applicability.....	3
2. Summary of Method.....	3
3. Definitions.....	3
4. Personnel Qualifications and Responsibilities	4
5. Equipment and supplies	4
6. Procedures	4
7. Quality Control and Quality Assurance	12
8. References.....	13
Appendix A. Guidelines Managing Electronically-stored Sensitive Information.....	19
Appendix B. Purdue University Password Requirements	20
Appendix C. Electronic Data Transfer Form	21

1. Scope and Applicability

- 1.1. Careful management of open-source, weather, and lagoon-characterization data is required to ensure that these data are all accurately documented and preserved.
 - 1.1.1. Data generated with the Tunable Diode Laser Absorption Spectrometer (TDLAS) is needed to properly apply the radial plume mapping method, and thus to document the changing ammonia concentrations of the air surrounding the open-air source.
 - 1.1.2. Data generated by Ultraviolet Differential Optical Absorption Spectroscopy (UV-DOAS) is needed to properly apply the backward Lagrangian Stochastic emissions model, and thus to document the changing ammonia and hydrogen sulfide concentrations of the air surrounding the open-air source
 - 1.1.3. Data generated by INNOVA Model 1412 Photoacoustic Multi-gas Monitor (SOP G7) to quantitate total and individual volatile organic compounds (VOCs) from the air surrounding the open-air source needs to be properly documented.
 - 1.1.4. Data on meteorological and lagoon conditions are needed for valid modeling of the effect of weather and microclimate on emissions.
- 1.2. The data management regime must be capable of managing data collected during multiple measurement periods, and at multiple study locations.

2. Summary of Method

The objective of data management is to ensure data security, easy data identification and retrieval, and efficient publication. All project-related files and data must be classified and organized in the computerized research data storage system. The tools to deliver, backup, inspect, analyze and present data must be available and kept current with the latest updates.

3. Definitions

- | | |
|-------------------|--|
| 3.1. bLS | Backward Lagrangian Stochastic |
| 3.2. CD | Compact disk |
| 3.3. DM | Data Manager |
| 3.4. DVD | Digital video disk |
| 3.5. Flash memory | Non-volatile rewritable memory |
| 3.6. FOS | Field operations staff |
| 3.7. HD | Computer hard drive |
| 3.8. ODBC | Open data base connectivity |
| 3.9. PAAQL | Purdue Agricultural Air Quality Laboratory |
| 3.10. PAML | Purdue Applied Meteorology Laboratory |
| 3.11. PIC | Path-integrated concentration |
| 3.12. PM | Project Manager |
| 3.13. ppb | Parts per billion (by volume) |
| 3.14. QAM | Quality Assurance Manager |
| 3.15. QAPP | Quality Assurance Project Plan |

3.16. RPM	Radial Plume Mapping method of emissions measurement
3.17. Site PI	Site Principal Investigator
3.18. SMP	Site monitoring plan
3.19. LAN Server	Local Area Network Sever computer
3.20. TDLAS	Tunable Diode Laser Absorption Spectrometer/Spectroscopy
3.21. UV-DOAS	Ultraviolet Differential Optical Absorption Spectrometer/ Spectroscopy
3.22. VOC	Volatile organic compounds
3.23. WXL	Weather and lagoon

4. Personnel Qualifications and Responsibilities

- 4.1. Most of these procedures assume a familiarity with the use of personal computers and general computer application software (e.g. MS Office), as well as the following:
 - 4.1.1. UV-DOAS procedures (SOP C1) and the WINDTRAX bLS software (SOP O1)
 - 4.1.2. The RPM software (SOP O2) and the TDLAS procedures (SOP C2)
 - 4.1.3. INNOVA 1412 Photoacoustic Multi-gas Monitor procedure (SOP G7)
 - 4.1.4. The data analysis software programs which are used to perform the transformations described in SOPs W1-W5
 - 4.1.5. The data acquisition and control software described in SOP W6
- 4.2. The site PI will follow an orderly system of data recording, organization, and safeguards, and will ensure that all members of the research team (including appropriate administrative personnel) understand and follow the system.
- 4.3. Data access management will be controlled according to Purdue University (Information Technology at Purdue) procedures defined for sensitive data (Appendix A). Those authorized to access research data will use strong passwords that are difficult to crack, according to Appendix B. Passwords will not be written down or shared with others. Passwords will be changed on a regular basis (every two months at a minimum).
- 4.4. Only those people explicitly authorized to access the research data will be allowed to write to the files (Appendix A).

5. Equipment and supplies

- 5.1. Data transfer pouch
- 5.2. UV-DOAS 4-Gb flash memory drives (2 per measurement period)
- 5.3. 64Mb CompactFlash® memory card (1 per measurement period)
- 5.4. 256Mb flash memory sticks (3 per measurement period)
- 5.5. Blank CDs
- 5.6. Fire-proof data vault
- 5.7. Innova Gas Monitor Software (Version 7304)

6. Procedures

- 6.1. Classification of electronic files
 - 6.1.1. Classify all electronic files according to the following four categories:

- 6.1.1.1. Research information and data. These files, if lost, cannot be rebuilt or recovered.
 - 6.1.1.1.1. Measurement data
 - 6.1.1.1.2. Data acquisition configuration data
 - 6.1.1.1.3. Calibration data forms
 - 6.1.1.1.4. Data logged by instruments (TDLAS and UV-DOAS) with internal data storage
 - 6.1.1.1.5. Calibration data recorded by TDLAS and UV-DOAS systems, and electronic calibration forms
 - 6.1.1.1.6. Data recorded by INNOVA 1412 Photoacoustic multi-gas monitor
 - 6.1.1.1.7. Field notes
 - 6.1.1.1.8. Email messages
 - 6.1.1.1.9. Image files (digital photos or graphs)
- 6.1.1.2. Project-specific configurations that could be rebuilt or recovered, if lost
 - 6.1.1.2.1. Hardware configuration files
 - 6.1.1.2.2. Configurations of firewalls, virus protection, etc
 - 6.1.1.2.3. Email accounts, address books, message rules
 - 6.1.1.2.4. Favorite lists
- 6.1.1.3. Project-specific software and documents
 - 6.1.1.3.1. Thunder Scientific WINDTRAX
 - 6.1.1.3.2. Boreal Laser GasView2
 - 6.1.1.3.3. Arcadis RPM
 - 6.1.1.3.4. INNOVA Gas Monitor Software 7304
 - 6.1.1.3.5. Campbell Scientific LoggerNet data logger programming and collection software
 - 6.1.1.3.6. National Instruments LabVIEW
 - 6.1.1.3.7. Files provided by vendors, such as instrument manuals and application notes
 - 6.1.1.3.8. Instrument communication software
- 6.1.1.4. General PC Software
 - 6.1.1.4.1. Microsoft Windows XP
 - 6.1.1.4.2. Microsoft Office
 - 6.1.1.4.3. WS_FTP server and ftp encryption software
 - 6.1.1.4.4. PC Anywhere
 - 6.1.1.4.5. Network Associates Enterprise
 - 6.1.1.4.6. Spybot S&D
 - 6.1.1.4.7. Genie Backup Manager
- 6.2. Data acquisition of UV-DOAS PIC spectra (SOP O1) for NH₃ and H₂S concentration calculation is conducted by the on-site 'bLS' computer (Fig. 1).
 - 6.2.1. Data is stored in a 4Gb flash memory drive on each UV-DOAS receiver. After 20 days, memory used is approximately 500Mb.
- 6.3. Data acquisition of TDLAS PIC spectra (SOP O2) for NH₃ concentration calculations is conducted by each of the on-site 'TDLAS/scanner' computers (Fig. 2).
 - 6.3.1. Data is stored in the HD of each TDLAS/scanner computer. After 20 days, memory used is approximately 3.3Mb.

- 6.4. Data acquisition using INNOVA 1412 Photoacoustic multi-gas monitor (SOP G7) for total and individual VOC concentrations stored directly in the HD of the LAN computer (Fig. 3).
- 6.5. Data acquisition of lagoon temperature, pH, and redox state (SOPs L1, L2 and L3), barometric pressure, air temperature, relative humidity, solar radiation, surface wetness, and data logger power supply voltage (SOPs E3, W1, W3, and W5) is conducted by a datalogger (SOP W6) (Fig. 4).
 - 6.5.1. Data is stored in a first in first out (FIFO) memory, and on a 64Mb CompactFlash® memory card. After 20 days, memory used is approximately 64Kb.
- 6.6. Data acquisition of wind components at three heights (SOP W2) is conducted by the on-site LAN Server computer (Fig. 4).
- 6.7. Data processing for the parameters listed in Section 6.2 is conducted by the on-site 'bLS' computer, which is connected to the UV-DOAS system by radio communications (Fig. 1).
- 6.8. Data processing for the parameters listed in Section 6.3 is conducted by the on-site LAN server computer, which is connected to the two 'TDLAS/scanner' computers by Ethernet (Fig. 2).
- 6.9. Data processing for the parameters listed in Section 6.4, and control of the INNOVA Multi-gas Monitor, are conducted by the Gas Monitoring Software 7304 running in the on-site LAN Server computer, which is connected by RS 232 cable to the INNOVA 1412 (Fig. 3).
- 6.10. Data processing for the parameters listed in Section 6.5 is conducted by the on-site Network Server computer, which is connected to the datalogger by radio communications.
- 6.11. File organization
 - 6.11.1. All project-specific files on computers will be in folders called *YYfarmn*, where *YY* is a two-letter abbreviation for the state and *n* is the farm identifier within the state. For example, two farms in Missouri would be identified as MOfarm1 and MOfarm2.
 - 6.11.2. The flash memory drive of each UV-DOAS instrument (Fig. 1) contains:
 - 6.11.2.1. Raw 5-min average PIC spectra [MinMinHrHr, PIC NH₃, PIC H₂S]
 - 6.11.2.2. Spreadsheet of 5-min PIC averages (filename 'C'MMDDYY [MinMinHrHr] containing: background PIC, SignalStrength, IntegrationTime, PIC NH₃, NH₃ measurement error, PIC H₂S, H₂S measurement error, RMSnoise)
 - 6.11.3. The datalogger flash memory (Fig. 4) contains:
 - 6.11.3.1. Raw data files (named by MMDDYY, containing farm number, MMDDYY, HrHrMinMin, 5-min averages of lagoon temperature, pH, and redox state, barometric pressure, air temperature, relative humidity, solar radiation, surface wetness, and power supply voltage) (SOP W6)
 - 6.11.4. In the on-site computer associated with both UV-DOAS systems (Fig. 1), termed the 'bLS computer', store all project date-specific measurement files in folders called MMDDYY where MM is numerical month, DD is numerical day in month, and YY is a two-letter abbreviation for the year in which the WINDTRAX bLS program was started. Organize the subfolders as:
 - 6.11.4.1. Program. WINDTRAX bLS software
 - 6.11.4.2. Config. Contains configuration files for bLS at location *YYfarmn*.

- 6.11.4.3. Touchdown tables (See SOP O1) for *YYfarmn*
- 6.11.4.4. 5-min 2-m height wind turbulence statistics (SOP W2) for *YYfarmn* so that statistics coincide with periods of PIC measurements according to SOP O1.
 - 6.11.4.4.1. Wind data is transferred from the LAN server computer to the UV-DOAS systems through the Ethernet connection.
- 6.11.4.5. UV-DOAS Instrument 1 data for location *YYfarmn*
 - 6.11.4.5.1. Raw 5-min average PIC spectra [MinMinHrHr, PIC NH₃, PIC H₂S
 - 6.11.4.5.2. Spreadsheet of 5-min PIC averages (filename 'C'MMDDYY [MinMinHrHr] containing: background PIC, SignalStrength, IntegrationTime, PIC NH₃, NH₃ measurement error, PIC H₂S, H₂S measurement error, RMS noise)
- 6.11.4.6. UV-DOAS Instrument 2 data for location *YYfarmn*
 - 6.11.4.6.1. Raw 5-min average PIC spectra [MinMinHrHr, PIC NH₃, PIC H₂S
 - 6.11.4.6.2. Spreadsheet of 5-min PIC averages (filename 'C'MMDDYY [MinMinHrHr] containing: background PIC, SignalStrength, IntegrationTime, PIC NH₃, NH₃ measurement error, PIC H₂S, H₂S measurement error, RMSnoise)
- 6.11.4.7. WINDTRAX input spreadsheet for location *YYfarmn*
 - 6.11.4.7.1. 5-min average difference in PICs and wind data (filename MMDDYY [MinMinHrHr]; containing: turbulence statistics, PIC NH₃, PIC H₂S)
- 6.11.4.8. WINDTRAX output file for location *YYfarmn* (filename MMDDYY [MinMinHrHr])
- 6.11.5. In the on-site data acquisition computer associated with each TDLAS/scanner system (each of 2 computers and associated systems, located according to Fig. 2), The two TDLAS/scanner systems are designated TDLAS1 and TDLAS2. All date-specific measurement files are in folders called MMDDYY where MM is numerical month, DD is numerical day in month, and YY is a two-letter abbreviation for the year in which the RPM program was started. Organize the subfolders as:
 - 6.11.5.1. Config1. Contains all files associated with first plane of measurement for RPM analysis of TDLAS
 - 6.11.5.1.1. Config. Contains geometry configuration file
 - 6.11.5.1.2. Input file. Includes PIC values for each time period of averaging
 - 6.11.5.1.2.1. Format according to SOP O2.
 - 6.11.5.1.3. Plume map graphs. Contains each averaging period plume map from the RPM program (See SOP O2) as a jpeg file (70Kb/image)
 - 6.11.5.1.4. Flux output file. Contains flux measurements with QA/QC checks on the RPM measurement according to SOP O2
 - 6.11.5.1.5. Valid raw wind data
 - 6.11.5.1.5.1. Format according to SOP W2.
 - 6.11.5.1.5.2. Data is transferred from the LAN server computer through the Ethernet connection.
 - 6.11.5.1.6. Parsed wind data so that the measurement period of wind data coincides with the measurement period of each complete cycle of PIC measurements.
 - 6.11.5.1.7. Raw PIC measurements from TDLAS
 - 6.11.5.1.7.1. Beam1. PIC values along beamline 1 with QA/QC flags (SOP C2).
 - 6.11.5.1.7.2. Beam2. PIC values along beamline 2 with QA/QC flags (SOP C2).

- 6.11.5.1.7.3. Beam3. PIC values along beamline 3 with QA/QC flags (SOP C2).
- 6.11.5.1.7.4. Beam4. PIC values along beamline 4 with QA/QC flags (SOP C2).
- 6.11.5.1.7.5. Beam5. PIC values along beamline 5 with QA/QC flags (SOP C2).
- 6.11.5.2. Config2. Contains all files associated with second plane of measurement for RPM analysis of TDLAS
 - 6.11.5.2.1. Config. Contains geometry configuration file
 - 6.11.5.2.2. Input file. Includes PIC values for each time period of averaging
 - 6.11.5.2.2.1. Format according to SOP O2.
 - 6.11.5.2.3. Plume map graphs. Contains each averaging period plume map from the RPM program (SOP O2) as a jpeg file (70Kb/image)
 - 6.11.5.2.4. Flux output file. Contains flux measurements with QA/QC checks on the RPM measurement according to SOP O2
 - 6.11.5.2.5. Valid raw wind data
 - 6.11.5.2.5.1. Format according to SOP W2.
 - 6.11.5.2.5.2. Data is transferred from the LAN server computer
 - 6.11.5.2.6. Parsed wind data so that the measurement period of wind data coincides with the measurement period of each complete cycle of PIC measurements
 - 6.11.5.2.7. Raw PIC measurements from TDLAS
 - 6.11.5.2.7.1. Beam6. PIC values along beamline 6 with QA/QC flags (SOP C2).
 - 6.11.5.2.7.2. Beam7. PIC values along beamline 7 with QA/QC flags (SOP C2).
 - 6.11.5.2.7.3. Beam8. PIC values along beamline 8 with QA/QC flags (SOP C2).
 - 6.11.5.2.7.4. Beam9. PIC values along beamline 9 with QA/QC flags (SOP C2).
 - 6.11.5.2.7.5. Beam10. PIC values along beamline10 with QA/QC flags (SOP C2).
- 6.11.6. In the on-site INNOVA data acquisition computer (which is also the on-site LAN server) organize the subfolders in the C drives as :
 - 6.11.6.1. C:\INNOVA (for all single point data acquisition and control)
 - 6.11.6.2. \Data acquisition program (Gas Monitoring Software7304)
 - 6.11.6.3. \Configuration file (common for all measurement locations)
 - 6.11.6.4. Data collected from the Gas Monitor Software 7304 (SOP G7) by the LAN server will be saved in the directory [C:\INNOVA\Farm number\MMDDYY\ODBC] (SOP G7).
 - 6.11.6.5. 5-min raw sample values will be stored on the LAN server in MS-access format (ODBC).
 - 6.11.6.6. Data includes: Measurement times, measurement sequence numbers, Error and user flags, atmospheric pressure, water vapor, NH₃ concentration, acetic acid concentration, methanol concentration, ethanol concentration.
- 6.11.7. In the on-site weather data acquisition computer (which is also the on-site LAN Server) organize the subfolders in the C drive as:
 - 6.11.7.1. LabVIEW files (for wind sensor data acquisition and control)
 - 6.11.7.1.1. Data acquisition program
 - 6.11.7.1.2. Configuration file (common for all measurement locations)
 - 6.11.7.1.3. Wind data files for YYfarm*n* (See SOP W2)
 - 6.11.7.1.3.1. File with 30 min of raw 16-Hz data for spectral analysis (including U, V, W, T_v)
 - 6.11.7.1.3.2. 5-min average statistics

- 6.11.7.1.3.3. 30-min average statistics
- 6.11.7.2. LoggerNet® datalogger software directories and subdirectories according to LoggerNet manual (page 2-3) for data collection and datalogger control of weather and lagoon characterization sensors (Section 5.2 of manual).
 - 6.11.7.2.1. Communication logs
 - 6.11.7.2.2. System. Includes binary files, configuration files and raw data downloads collected by the data acquisition program named as [MMDDYY-YYfarm*n*]
 - 6.11.7.2.3. PakBusGraph. Contains daily plots of raw data.
 - 6.11.7.2.3.1. Data files for YYfarm*n* (named by date MMDDYY, containing HrHrMinMin, 5-min averages of lagoon temperature, pH, and redox state, barometric pressure, air temperature, relative humidity, solar radiation, surface wetness, and power supply voltage)
- 6.11.7.3. Datalogger configuration and program editors
- 6.11.7.4. Library directory containing compilers and definition files
- 6.11.7.5. Calibration. Contains calibration sheets, gas cylinder logs, control charts, and other records
- 6.11.7.6. QAPP. The latest copy of the Quality Assurance Project Plan
- 6.11.7.7. SOP. Latest copies of all relevant Standard Operating Procedures
- 6.11.7.8. SMP. Site monitoring plan
- 6.11.7.9. Certifications
- 6.11.7.10. Measurement period records. Indicates dates and locations
- 6.11.7.11. Field Notes
 - 6.11.7.11.1. Typed logs of activities and observations (All notes must include name and date of the person making the note)
 - 6.11.7.11.2. Producer Event forms and updated farm specific information (layout drawings etc) other farm lagoon management data (information on animals, weight, feed, etc) collected from the farm manager (SOP S7)
- 6.11.7.12. Photos. Digital pictures of field setup and observations
- 6.11.7.13. Email. Contains email messages and attachments. Name the email account yyfarm*n*@zzz.zzz where zzz.zzz is the internet service provider's domain. Outlook Express is the preferred email program.
- 6.11.7.14. Miscellaneous
 - 6.11.7.14.1. Site equipment inventory including gas cylinders, etc
 - 6.11.7.14.2. Transported equipment inventory
- 6.11.8. The administrative server at PAML contains the following folders and information, and operates various personal computers:
 - 6.11.8.1. Weather data from outside sources for each day and every location that measurements are taken. Example of an outside source might be; <http://www.wunderground.com/us/nc/elizabethtown.html>.
 - 6.11.8.2. Data ftped from YYfarm*n*
 - 6.11.8.3. UV-DOAS and bLS data and configuration files
 - 6.11.8.4. TDLAS and RPM data and configuration files
 - 6.11.8.5. Excel files of text data exported from bLS and RPM directories during processing
 - 6.11.8.6. LoggerNet configuration files

- 6.11.8.7. INNOVA Gas Monitor Software 7304 configuration files
 - 6.11.8.8. LabVIEW configuration files
 - 6.11.8.9. Excel files of data exported from LoggerNet during processing
 - 6.11.8.10. Excel files of data exported from LabVIEW during processing.
 - 6.11.8.11. AutoCad drawings of lagoon and equipment
 - 6.11.8.12. Data tables for reports and manuscripts
 - 6.11.8.13. Farm production data
 - 6.11.8.14. Reports
 - 6.11.8.15. ftp transfer logs
- 6.12. Data security
- 6.12.1. The on-site computer associated with the UV-DOAS (Fig. 1) is linked through an Ethernet local area network to the Network Server computer.
 - 6.12.2. Each on-site data acquisition computer linked with each TDLAS instrument is linked to the Network server computer in the trailer through an Ethernet local area network (SOP U4).
 - 6.12.3. Datalogger
 - 6.12.3.1. Three 5-digit security passwords are needed to change the data acquisition program, download data, or change the clock.
 - 6.12.3.1.1. FOS will each have all security codes.
 - 6.12.3.2. Replace 1Mb flash memory at the end of each measurement period (identified here as WXL).
 - 6.12.3.2.1. Put flash memory into the zippered Data pouch.
 - 6.12.4. LoggerNet will be operated as a service on the on-site computer, so that the program will restart after power failures.
 - 6.12.5. Innova Gas Monitoring Software 7304 shortcut will be placed in the start up folder so the program will restart after power failure or restart.
 - 6.12.6. Program Windows “Scheduled Tasks” to ftp the 24-h data files from the on-site weather data acquisition computer, Innova Gas Monitoring software, and the data acquisition computers linked to the TDLAS instruments and UV-DOAS instruments to the PAML computer at midnight.
 - 6.12.6.1. All ftp file transfers are encrypted.
 - 6.12.7. Collect archived raw data from flash drives in each UV-DOAS instrument (approximately 500MB of data on each of the 4GB flash drives) at the conclusion of the measurement period.
 - 6.12.7.1. Replace the flash drive in each instrument.
 - 6.12.7.2. Copy content of each UV-DOAS flash drive onto two CDs.
 - 6.12.7.2.1. Identify CD with location (*YYfarm*n**), measurement period (MM-DD-YY to MM-DD-YY), “UVDOAS1” for “UVDFlash1” and “UVDOAS2” for “UVDFlash2” and label “vault” or “carry”.
 - 6.12.7.2.2. Copy using LAN Server computer
 - 6.12.7.2.3. Put one copy of each of the two CDs into the fire-proof data vault in the trailer.
 - 6.12.7.2.4. Put the other copy of each CD in Data pouch.
 - 6.12.7.3. Store all data-containing UV-DOAS Flash drives in Data pouch.

6.12.8. Field notes

6.12.8.1. Notes are stored on the HD of the LAN server computer, and are (automatically) backed up daily on an external HD.

6.12.8.2. Email field notes to PM and DM at the end of each work day in the field, before leaving the site.

6.12.8.3. Download field notes onto flash memory stick.

6.12.8.4. Store memory stick in the zippered Data pouch.

6.12.9. FOS copies of calibration data and control charts

6.12.9.1. Email to PM, DM, and QAM after each 20-day measurement period.

6.12.9.2. Retain one paper copy in trailer.

6.12.9.3. Download onto flash memory stick with field notes.

6.12.9.4. Store memory stick in the zippered Data pouch.

6.12.10. Send (by ftp) configuration files to DM whenever they are updated.

6.12.11. File backups by field staff

6.12.11.1. Back up project folders and subfolders *YYfarm*n** of each onsite computer onto an external hard drive at 1:00 a.m. each night during the measurement period.

6.12.11.2. Back up data files onto DVD at the conclusion of every measurement period.

6.12.11.2.1. Two copies each of UVDOAS1, UVDOAS2 from LAN server.

6.12.11.2.2. Two copies each of TDLAS 1 and TDLAS 2 from respective Data Acquisition computers.

6.12.11.2.3. Two copies of INNOVA data files from LAN server

6.12.11.2.4. Two copies of WXL data from LAN server

6.12.11.2.5. Identify each CD with data type (above), location (*YYfarm*n**) and measurement period (MM-DD-YY to MM-DD-YY). Label one copy of each DVD as “vault”, one copy as “carry”.

6.12.11.2.6. Put “vault” copy of DVD into the fire-proof data vault.

6.12.11.2.7. Put “carry” copy of DVD in the data pouch.

6.12.11.3. Manually back up the operating system of any computer onto an external HD whenever significant changes are made to the system.

6.12.12. Automatically back up filesystem of the LAN server onto an external HD every day at 2:00 a.m.

6.12.13. Carry Data pouch in hand luggage upon returning to PAML.

6.12.13.1. Data pouch includes:

6.12.13.1.1. For latest measurement period:

6.12.13.1.1.1. One pair of 4-Gb UV-DOAS flash memory drives (UVDFlash1 and UVDFlash2)

6.12.13.1.1.2. One pair of CDs with copies of UV-DOAS flash memory drives (UVDOAS1 and UVDOAS2)

6.12.13.1.1.3. Two CDs with TDLAS data (TDLAS1 and TDLAS2)

6.12.13.1.1.4. One CD with weather, and lagoon (WXL) and INNOVA data from recently-completed measurement period

6.12.13.1.1.5. One 64-Mb flash memory chip of weather and lagoon (WXL) data from recently-completed measurement period

6.12.13.1.1.6. One 256-Mb memory stick with Field notes, calibration data and control charts from recently-completed measurement period

- 6.12.13.1.1.7. One DVD with *YYfarmn* backup of all *YYfarmn* directories
- 6.12.13.1.2. For previous measurement period (material previously stored in data vault):
 - 6.12.13.1.2.1. One pair of CDs with copies of UV-DOAS flash memory drives (UVDOAS1 and UVDOAS2)
 - 6.12.13.1.2.2. Two CDs with TDLAS data from the previous measurement period (TDLAS1 and TDLAS2)
 - 6.12.13.1.2.3. One CD with weather, lagoon (WXL) and INNOVA data from previous measurement period
 - 6.12.13.1.2.4. One DVD with *YYfarmn* backup of all *YYfarmn* directories
- 6.12.13.1.3. Electronic data transfer forms for both recently-completed and previous measurement period (Appendix C) with upper section filled out.
- 6.12.13.2. Deliver Data pouch to DM on arrival.
 - 6.12.13.2.1. Document delivery of data to DM with Electronic Data Transfer Forms (Appendix C).
 - 6.12.13.2.1.1. Forms to be stored in the folder marked 'Electronic data Transmittal records' under the control of the DM.
- 6.12.14. DM archives files on the PAML computer onto an external HD drive every day at 5:00 a.m.
- 6.12.15. DM downloads all flash memory to a separate directory on PAML computer for backup.
- 6.12.16. DM stores all received CDs in an off-campus location.
- 6.12.17. DM archives files on the PAAQL server., which is backed up daily on the Purdue University backup system.
- 6.13. Retention of data
 - 6.13.1. Keep all data files for at least six (6) years.

7. Quality Control and Quality Assurance

- 7.1. Verify that the following software are included in their respective computers' startup menus, for automatic recovery after power failure.
 - 7.1.1. LoggerNet, Gas Monitor Software 7304, and LabVIEW (in the on-site, LAN server, computer)
 - 7.1.2. WINDTRAX program (in the bLS computer)
 - 7.1.3. RPM program (in each TDLAS/scanner data acquisition computer)
- 7.2. Verify that all data acquisition computers log power failures, and that emails are sent to the QAM and DM upon restart.
- 7.3. Check field computer and relevant software, and take immediate measures to solve related problems, if the daily data file ftp, and/or emails with other files are not received.
- 7.4. Multiple means of irreplaceable data transfer to PAML
 - 7.4.1. Daily ftp of files
 - 7.4.2. Hand delivery of data from each measurement period at end of measurement period and at the end of the next sequential measurement period (stored in the data vault)
- 7.5. Raw data stored in multiple places prior to transfer to PAML
 - 7.5.1. UV-DOAS raw (Fig. 1)
 - 7.5.1.1. Flash memory drive of each UV-DOAS instrument

- 7.5.1.2. bLS computer HD
- 7.5.2. TDLAS raw and RPM processed data (Fig. 2)
 - 7.5.2.1. TDLAS data acquisition computer HD
 - 7.5.2.2. External HD
- 7.5.3. bLS computer filesystems (including wind measurements and bLS input files and results) (Fig. 1)
 - 7.5.3.1. bLS computer HD
 - 7.5.3.2. External HD
- 7.5.4. INNOVA raw data (Fig. 3)
 - 7.5.4.1. LAN server HD
 - 7.5.4.2. External HD
- 7.5.5. Datalogger raw data (excluding wind measurements) (Fig. 4)
 - 7.5.5.1. CompactFlash memory card
 - 7.5.5.2. On-site datalogger
 - 7.5.5.3. LAN server HD
 - 7.5.5.4. External HD
- 7.5.6. LAN Server computer filesystems (including wind measurements) (Figs. 1-4)
 - 7.5.6.1. HD of on-site computer
 - 7.5.6.2. External HD
- 7.6. DM tracks all modifications to electronic files. This includes tracking the original data entered, tracking who and when an individual or system viewed or changed any data, and recording why data was changed.
 - 7.6.1. Save and archive all raw data in their original form.
 - 7.6.2. Use a “notes” worksheet in Excel files to describe modifications.
 - 7.6.3. Include date at the end of file name in the following format: MM-DD-YY, ex: 01-09-06. Leading zeros in day and month can be omitted, ex. 1-9-06.
 - 7.6.4. Utilize electronic audit trails as much as possible.

8. References

- 8.1. Campbell Scientific Inc. 2005. LoggerNet Instruction Manual, v. 3.2, Campbell Scientific Inc., Logan UT.
- 8.2. Campbell Scientific Inc. 2006. CR1000 Measurement and Control System Operators Manual. v. 3/06, Campbell Scientific Inc., Logan, UT
- 8.3. Information Technology at Purdue. 2006. Electronically Stored (Computer-based) Information. Available at:
<http://www.itap.purdue.edu/security/procedures/dataHandling/electrStored.cfm>. Accessed 2-28-2006.
- 8.4. Information Technology at Purdue. 2006. Password Guidelines. Available at:
<http://www.itap.purdue.edu/security/procedures/passGuidelines.cfm>. Accessed 2-28-2006.
- 8.5. SOP C1. 2006. Open Path Measurement of Ammonia and Hydrogen Sulfide with the Cerex UV Sentry Ultraviolet Differential Optical Absorption Spectrometer. Standard Operating Procedure C1. Purdue Applied Meteorology Lab.

- 8.6. SOP C2. 2006. Measurement of Ammonia with the Boreal Laser GasFinder Tunable Diode Laser Absorption Spectrometer (TDLAS) Standard Operating Procedure C2. Purdue Applied Meteorology Lab.
- 8.7. SOP E3. 2006. The Measurement of Solar Radiation with the Licor Model 200SL or 200SZ Silicon Pyranometer. Standard Operating Procedure E3. Purdue Ag Air Quality Lab.
- 8.8. SOP G7. 2006. Use of the INNOVA 1412 Photoacoustic Multi-gas Monitor. Standard Operating Procedure G7. Purdue Ag Air Quality Lab.
- 8.9. SOP L1. 2006. Measurement of Lagoon pH with Innovative Sensors Model CSIM11 Sensor. Standard Operating Procedure L1. Purdue Applied Meteorology Lab.
- 8.10. SOP L2. 2006. Measurement of Lagoon Temperature with Campbell Scientific Model 107-L Thermistor. Standard Operating Procedure L2. Purdue Applied Meteorology Lab.
- 8.11. SOP L3. 2006. Measurement of Lagoon Redox State with Campbell Scientific CSIM11_ORP Sensor. Standard Operating Procedure L3. Purdue Applied Meteorology Lab.
- 8.12. SOP O1. 2006. Emissions Estimation Using the Thunder Beach Scientific Windtrax Backward Lagrangian Stochastic Model. Standard Operating Procedure O1. Purdue Applied Meteorology Lab.
- 8.13. SOP O2. 2006. Emissions Estimation Using the ARCADIS Radial Plume Mapping Model. Standard Operating Procedure O2. Purdue Applied Meteorology Lab.
- 8.14. SOP S7. 2006. Producer Collaborations at Area Source Monitoring Sites. Standard Operating Procedure S7. Purdue Applied Meteorology Lab.
- 8.15. SOP U4. 2006. Open-source Instrument Trailer. Standard Operating Procedure U4. Purdue Applied Meteorology Lab.
- 8.16. SOP W1. 2006. Measurement of Atmospheric Temperature and Humidity with the Vaisala Model HMP45C Sensor and Solar Shield. Standard Operating Procedure W1. Purdue Applied Meteorology Lab.
- 8.17. SOP W2. 2006. Measurement of Wind with the RM Young Model 81000 3-Dimensional Sonic Anemometer. Standard Operating Procedure W2. Purdue Applied Meteorology Lab.
- 8.18. SOP W3. 2006. Measurement of Wetness with the Campbell Scientific Model Resistance Grid. Standard Operating Procedure W3. Purdue Applied Meteorology Lab.
- 8.19. SOP W5. 2006. The Measurement of Barometric Pressure with the Setra Model 278 (Campbell Scientific CS100) Barometer. Standard Operating Procedure W5. Purdue Applied Meteorology Lab.
- 8.20. SOP W6. 2006. Establishment, Data Acquisition and Control of Weather and Lagoon Characterization Hardware.. Standard Operating Procedure W6. Purdue Applied Meteorology Lab.

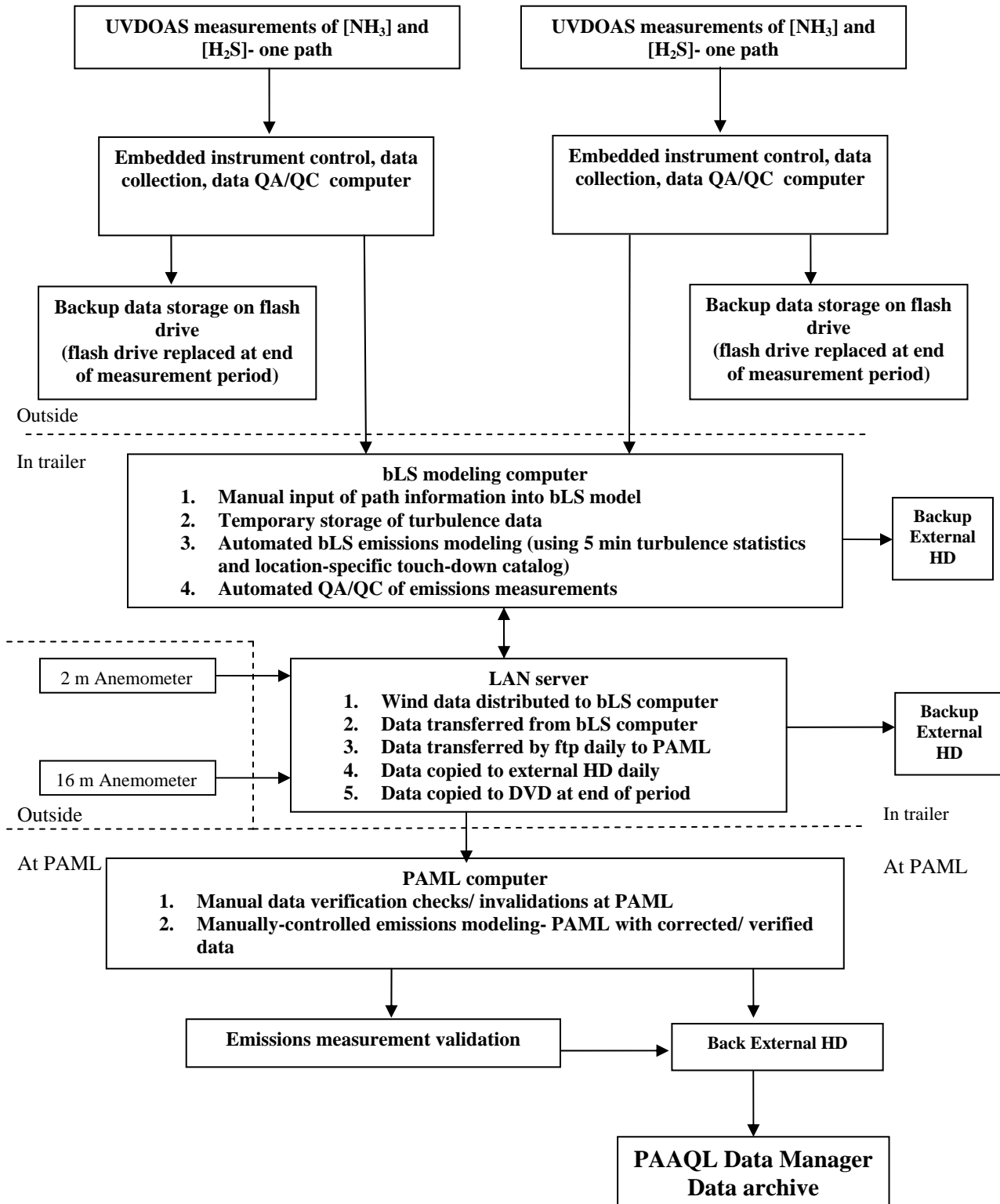
Figure 1. Data Processing and Management: UV-DOAS and bLS Data.

Figure 2. Data Processing and Management: TDLAS and RPM Data.

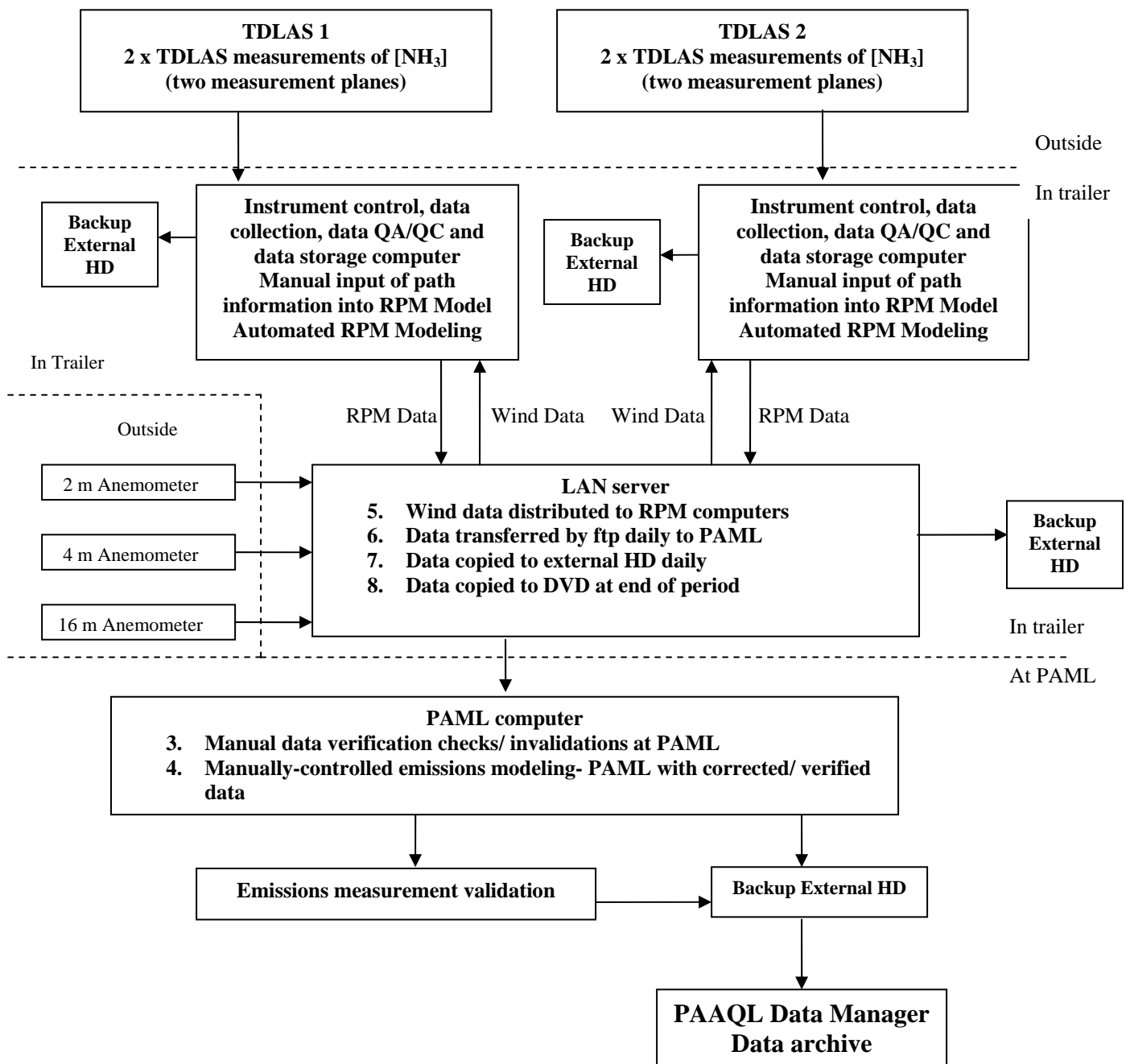


Figure 3. Data Processing and Management for INNOVA 1412.

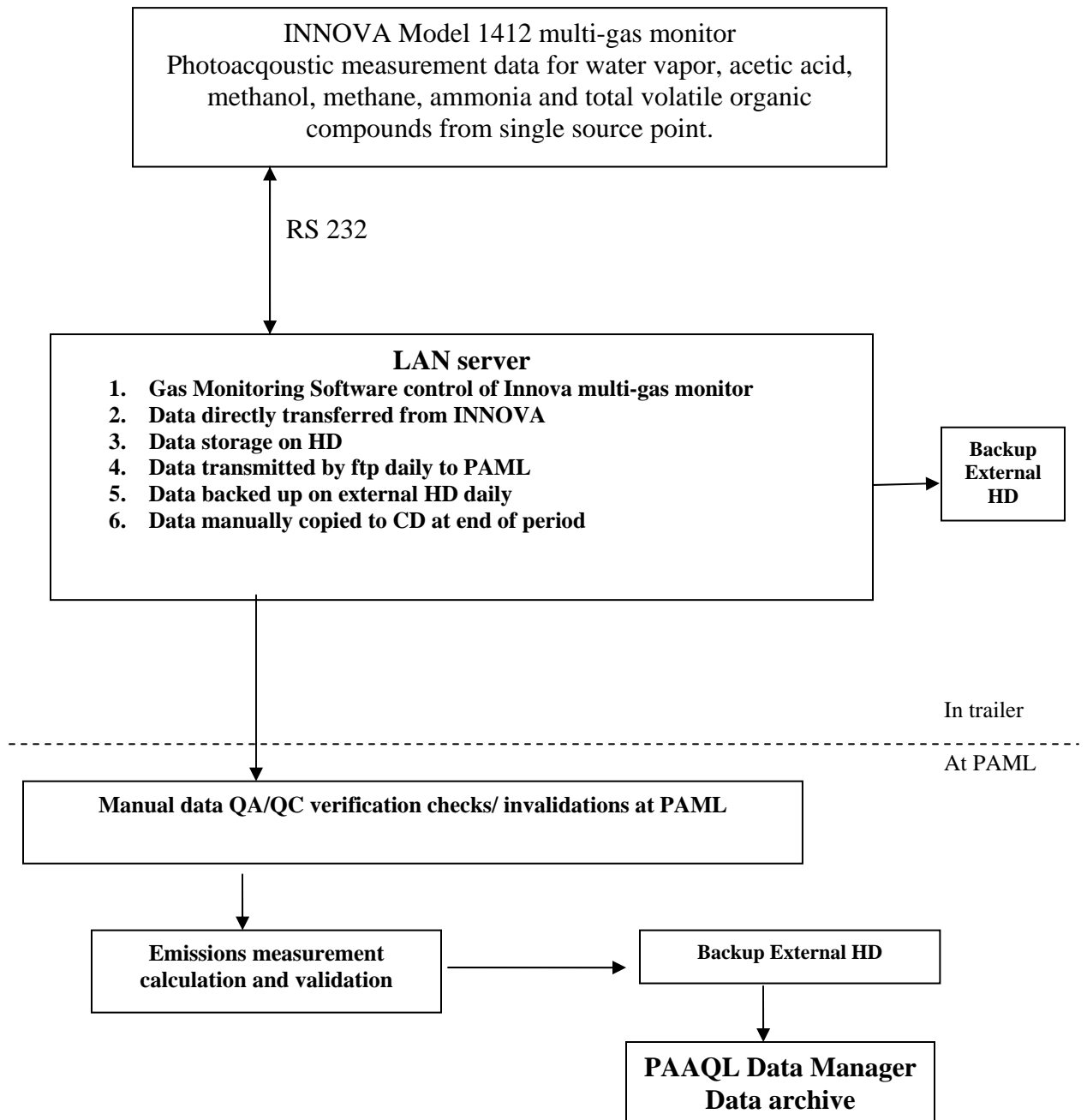
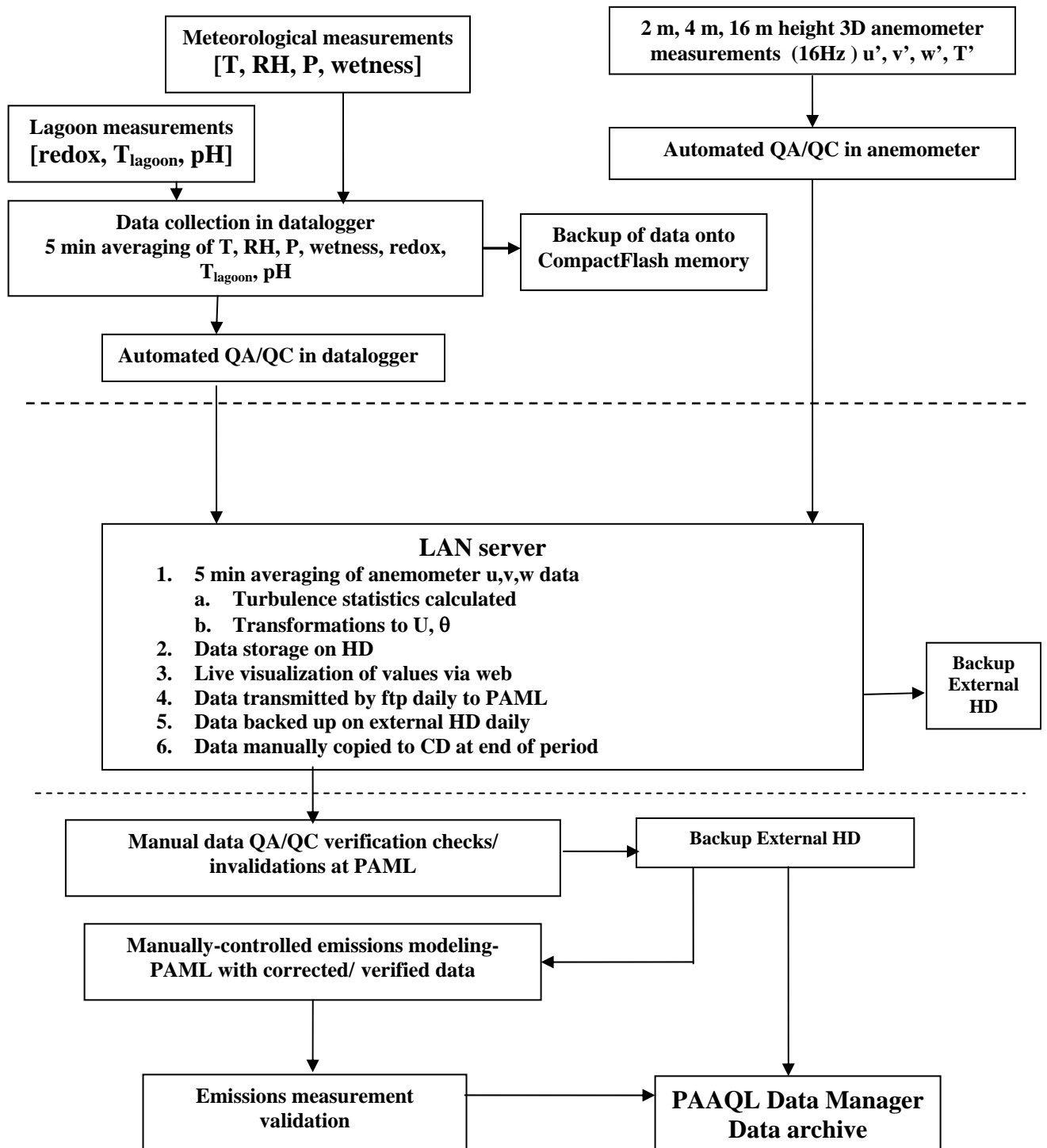


Figure 4. Data Processing and Management: Meteorological and Lagoon Data.



Appendix A

Guidelines for Managing Electronically-stored Sensitive Information¹

Action	Sensitive Information
Storage on fixed media with access controls	No encryption required
Storage on fixed media without access controls, but accessible via the web	Not advised. If you must store data via this media, it must be encrypted.
Storage on fixed media without access controls, but not accessible via the web	No encryption required
Storage on removable media	No encryption required
Read access to information (includes duplication)	Access is based on roles defined by QAPP
Create / Update access to information	Access is based on roles defined by QAPP
Delete access to information	Access is based on roles defined by QAPP
Print hard copy report of information	Unattended printing permitted only if physical access controls are used to prevent unauthorized viewing.
Internal labeling of information at the application or screen/display level	No special requirement
Disposal of the physical electronic media device (diskettes, tapes, hard disks, etc.)	Physical destruction beyond ability to recover.
Disposal of information	Delete files through normal platform delete command, option or facility.
Data Manager & Information Owner review Data Confidentiality for continued applicability	Review annually

1: modified from Information Technology at Purdue:

<http://www.itap.purdue.edu/security/procedures/dataHandling/electrStored.cfm>,

Appendix B

Purdue University Password Requirements

- Passwords must contain at least 1 letter.
- Passwords must contain at least 1 number or punctuation mark.
- Passwords must be at least 8 characters long.
- Passwords must contain more than 4 unique characters.
- Passwords may not contain easily guessed words (e.g. Purdue, itap, boiler).
- Passwords may not contain a name or parts of the name (e.g., Bill, Julie, Bob, or Susan).
- New passwords must be different than the previous password (re-use of the same password will not be allowed for 180 days).
- Passwords never should be stored on your computer or written down and stored in plain sight (e.g., taped to the bottom of your computer keyboard). If a password must be written down, it should be locked up and stored in a place that is difficult for others to access.
- Passwords may be used only by the authorized user. Do not share your password with anyone.
- All users must change their password at least once every 30 days.
- If you suspect your password has been compromised, it should be changed immediately.

Appendix C Electronic Data Transfer Form

Location of measurements: _____

Period represented: From Date (MM/DD /YYYY) _____ time (GMT: 24h clock) _____: _____

To Date (MM/DD/YYYY) _____ time (GMT: 24h clock) _____: _____

Data preparation/ copying done by: _____

Calibration conducted during period

UVDOAS1: s/n _____ Date (MM/DD/YYYY) _____ time (GMT: 24h clock) _____: _____

UVDOAS2: s/n _____ Date (MM/DD/YYYY) _____ time (GMT: 24h clock) _____: _____

TDLAS1: s/n _____ Date (MM/DD/YYYY) _____ time (GMT: 24h clock) _____: _____

TDLAS2: s/n _____ Date (MM/DD/YYYY) _____ time (GMT: 24h clock) _____: _____

Notes:

Data Receipt

Received: Date (MM/DD/YYYY) _____ time (GMT: 24h clock) _____: _____

Physical condition of TDLAS1 data CD (Excellent / Good / Fair / Poor)

Physical condition of TDLAS2 data CD (Excellent / Good / Fair / Poor)

Physical condition of UVDOAS1 data CD (Excellent / Good / Fair / Poor)

Physical condition of UVDOAS2 data CD (Excellent / Good / Fair / Poor)

Physical condition of WXL and INNOVA data CD (Excellent / Good / Fair / Poor)

Physical condition of WXL and INNOVA flash memory chip (Excellent / Good / Fair / Poor)

Physical condition of UVDFlash1 memory drive (Excellent / Good / Fair / Poor)

Physical condition of UVDFlash2 memory drive (Excellent / Good / Fair / Poor)

Physical condition of Backup DVD (Excellent / Good / Fair / Poor)

Physical condition of memory stick (Excellent / Good / Fair / Poor)

Received by: _____

**MEASUREMENT OF RELATIVE HUMIDITY (RH) AND TEMPERATURE
WITH THE NOVUS RHT-WM TRANSMITTER Standard Operating
Procedure (SOP) E2**

**MEASUREMENT OF RELATIVE HUMIDITY (RH) AND TEMPERATURE
WITH THE NOVUS RHT-WM TRANSMITTER
Standard Operating Procedure (SOP) E2**

Prepared by

Erin L. Cortus, Congna Li, and Bill W. Bogan

Reviewed by

Teng-Teoh Lim and Albert J. Heber

Effective Date: December 28, 2007

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability.....	3
2. Summary of Method.....	3
3. Definitions of Terms and Acronyms	3
4. Health and Safety.....	4
5. Interferences.....	4
6. Cautions.....	4
7. Personnel Qualifications	5
8. Equipment and Supplies	5
9. Procedures	5
10. Data and Records Management	10
11. Quality Control and Quality Assurance Section.....	10
12. References.....	11

1. Scope and Applicability

- 1.1. The NOVUS RHT humidity and temperature transmitter is used in indoor and outdoor environments to measure relative humidity (RH) and temperature (T).
- 1.2. The temperature measurement range of the instrument is -40°C to $+120^{\circ}\text{C}$. Accuracy is $\pm 0.5^{\circ}\text{C}$ at 25°C , with a maximum error of $\pm 2.0^{\circ}\text{C}$ between -40°C and 80°C . Barn temperatures may range between -10°C and 40°C , but are usually maintained at approximately 20°C , depending on the species and age of animals.
- 1.3. The RH measurement range is 0 to 100%, as compared with typical average barn RH of 65%. Accuracy is $\pm 1.5\%$ RH for 20 to 80%RH and $\pm 3.0\%$ for RH values below 20% and above 80% (at 25°C).
- 1.4. The NOVUS RHT transmitter can be calibrated with a series of salt solutions.
- 1.5. The NOVUS RHT transmitter can be calibrated or spot-checked with a fundamental measurement instrument or a NIST-transfer device.
- 1.6. The NOVUS RHT transmitter is available in wall mount (WM) or duct mount (DM) housing options. The sensor and electronics are identical between the two models; mounting instructions were provided in this SOP for the WM version only.

2. Summary of Method

The purpose of the method is to continuously monitor the RH and T of indoor and exhaust air. The NOVUS RHT humidity and temperature transmitter uses a microprocessor-based sensor. The thin polymer film either absorbs or releases water vapor as the relative humidity of the ambient air rises or drops. The dielectric properties of the film depend on the amount of water contained in it; as the RH changes, the capacitance of the sensor also changes. The electronic circuit of the instrument measures the capacitance of the sensor and converts it into a humidity reading. The sensor is protected from physical contact with solid, foreign objects greater than 1 mm in diameter by an IP40 capsule. The capsule should be covered with a filter material to further reduce the build-up of dust on the sensor element, although the sensor itself can be cleaned. The electronics in the transmitter are protected by IP65 housing. This makes the transmitter suitable for humid and wet environments, such as those found on livestock farms.

3. Definitions of Terms and Acronyms

- | | | |
|------|-------------------------|--|
| 3.1. | DAC | Data acquisition and control |
| 3.2. | K_2SO_4 | Potassium sulfate |
| 3.3. | LiCl | Lithium chloride |
| 3.4. | MgCl_2 | Magnesium chloride |
| 3.5. | NaCl | Sodium chloride |
| 3.6. | PAAQL | Purdue Agricultural Air Quality Laboratory |
| 3.7. | RH | Relative humidity |
| 3.8. | SOP | Standard Operating Procedure |
| 3.9. | T | Temperature |

4. Health and Safety

- 4.1. Be careful when working with the electrical power connections.
- 4.2. The lithium chloride (LiCl) solution used in the instrument calibration (Section 9.3) is corrosive so avoid contact with exposed skin. LiCl salt and solution are harmful if swallowed. Never add water directly to dry LiCl salt.

5. Interferences

- 5.1. Barn environments will deposit dust particles onto the RH/Temp sensor, which will likely decrease the accuracy of the RH sensors if they are not cleaned frequently enough.
- 5.2. High concentrations of chloride acid, nitride acid, sulfuric acid, ammonia, acetone, ethanol or propylene glycol may cause irreversible measurement errors. The concentration of ammonia gas that causes the sensor to shift has not been quantified, but the impact is assumed to be similar to the effects of ammonia on other electronics.
- 5.3. Extended exposure to contaminating vapors or high humidity may cause the RHT sensor calibration to change.

6. Cautions

- 6.1. Avoid installing a transmitter at a measurement location where the air is not well-mixed, or which is not representative of the barn area.
- 6.2. The NOVUS RHT transmitter comes factory-set with a temperature range of 0 to 100 °C. The temperature range can be adjusted to -40 to 120 °C using the TXConfig software. Ensure that the temperature range of the instrument set in AirDAC is the same as the current sensor settings.
- 6.3. When using the HMI41 humidity calibrator or equivalent for spot-checks and/or calibration, allow a sufficient amount of time (at least 10-30 min) for both the transmitter and calibration indicator to stabilize. Take several minutes' worth of data to compare, especially for RH measurement.
- 6.4. When using salt chambers to calibrate the RH transmitter, allow enough time (at least 10-30 min) for the transmitter to stabilize after the sensor probe is inserted into the salt calibration chamber. Other salt calibration procedures are as follows:
 - 6.4.1. Discard LiCl solutions that have been exposed to temperatures below +18°C. Exposure of the LiCl solution to temperatures below +18°C will cause a permanent change in its equilibrium humidity.
 - 6.4.2. Hold the probe as little as possible. Do not hold the salt chamber or other parts of the calibrator in your hand during calibration, as they may warm up and cause errors in the readings.
 - 6.4.3. Place the thermometers in holders when the thermometers are not in use, or when the calibrator is being moved from one place to another.
 - 6.4.4. Usually, errors during humidity calibration are due to temperature differences. Use the salt solutions in the part of the room where the temperature is most stable and keep it out of direct sunlight and away from localized heat sources.

7. Personnel Qualifications

- 7.1. Personnel should be trained in the use of the instrument before initiating the procedure.
- 7.2. Each analyst must read the entire instrument manual and this SOP before operating the instrument.

8. Equipment and Supplies

- 8.1. NOVUS RHT-WM Humidity Transmitter (Novus Electronics, Brazil)
- 8.2. Air conditioner filter material and cable ties, or equivalent.
- 8.3. Computer (if sensor configuration is required)
- 8.4. TXConfig software and Interfacing Cable (if sensor configuration is required)
- 8.5. Vaisala HMK15 Humidity Calibrator (Vaisala Inc., Woburn, MA) or equivalent set of salt solutions and calibration chamber.
 - 8.5.1. Certified salts to make standard salt solutions for HMK15 humidity calibrator or equivalent (LiCl salt for 11% RH, MgCl₂ salt for 33% RH, NaCl salt for 75% RH, and K₂SO₄ for 97% RH)
 - 8.5.2. Distilled deionized water
- 8.6. Vaisala HMI41 Portable RH/T transmitter with NIST-calibration certificate (Vaisala Inc., Woburn, MA) or equivalent NIST-calibrated device for RH and temperature comparisons
- 8.7. Spare NOVUS RHT-WM transmitter and/or sensor.

9. Procedures

- 9.1. Installation of NOVUS RHT-WM
 - 9.1.1. The NOVUS RHT-WM humidity and temperature transmitters are intended to be mounted directly on the wall with the probe pointing downwards to assure the specified accuracy and protect the sensor. Choose a location which is representative of the larger environment, or of the specific exhaust air stream to be measured.
 - 9.1.2. For wall-mounting, open the transmitter cover to access two mounting holes.
 - 9.1.3. For measuring RH/T of barn exhaust air stream, locate a representative location to position the transmitter. A convenient installation approach is to attach the transmitter to the TEOM PM₁₀ sampling inlet supporting rod atop the enclosure unit (SOP P1).
 - 9.1.4. Wrap the sensor capsule with some filtering material and secure the material with a cable tie. An example filter material is an air-conditioner filter.
- 9.2. Electrical connections
 - 9.2.1. Thread the power and signal cable through the cable gland.
 - 9.2.2. In the center of the transmitter are two jumper switches (labeled 4 and 5) that can be set to Position A or Position B, independently, depending on the desired output variables. For humidity and temperature outputs, set both jumper switches to Position A. The Output 1 signal will be temperature, and the Output 2 signal will be RH. For other output configurations, refer to the transmitter operating manual.

- 9.2.3. The circuit board has six terminals for wiring (Fig. 1). The terminals are clearly identified on the circuit board inside the transmitter. The output signal range is 0-10 VDC for an excitation of 18 to 30 VDC

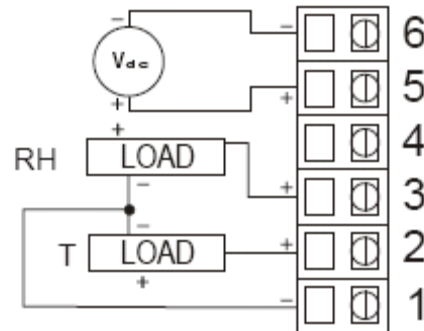


Figure 1. Wiring diagram of the RH/T transmitter. (Source: NOVUS RHT-WM and RHT-DM Datasheet).

- 9.2.1. Connect the power and ground conductors to terminals 5 and 6, respectively. Connect the digital signal conductor for humidity to terminal 3, the digital signal conductor for temperature to terminal 2, and a signal ground wire to terminal 1.
- 9.2.2. Connect the opposite end of the digital signal, power and ground wires to the appropriate terminals of the data acquisition and control (DAC) system (SOP B1).
- 9.2.3. Enter the sensor's signal and measurement ranges into the AirDAC software so that the RH/T readings can be converted, displayed, and recorded (SOP B2).
- 9.3. Calibration using the HWK15 Humidity Calibrator. Calibration is based on the fact that certain saturated salt solutions generate a specific, reproducible RH in the air above them. The salt solutions suitable for the HMK15 are lithium chloride LiCl (11% RH), magnesium chloride $MgCl_2$ (33% RH), sodium chloride NaCl (75% RH) and potassium sulphate K_2SO_4 (97% RH). Calibration using the HMK15 Humidity Calibrator is usually performed at a minimum of two different humidities to ensure the sensor accuracy over the entire humidity range (0-100 % RH). When calibrated with salt solutions, the accuracy of the sensor is $\pm 2\%$ RH (0-90% RH) and $\pm 3\%$ RH (90-100% RH).
- 9.3.1. Open the transit cover of the chamber. Remove the measurement cover from the chamber holder and press the transit cover on the holder.
- 9.3.2. Pour the required amount of deionized water (Table 1) into the chamber.
- 9.3.3. Sprinkle the contents of a salt package slowly into the chamber, stirring constantly. If using the measurement cup to transfer the salt, make sure that the cup is clean and dry. Rinse and dry the cup after every use. Amounts of salt added and final volumes of the standard solutions are listed in Table 1.

Table 1. Salt solutions used in HMK15 Humidity Calibrator.

Salt	Initial water added (mL)	Mass of salt (g)	Solution final volume (mL)
LiCl	14	15	18
MgCl ₂	3	30	30
NaCl	10	20	15
K ₂ SO ₄	10	30	20

- 9.3.4. Close the chamber with the chamber cover. Fasten the salt chamber on the base plate and close the measurement holes with rubber plugs. Make sure that chamber covers and plugs are carefully closed.
- 9.3.5. Write the preparation date of the solution on a sticker and affix to the chamber.
- 9.3.6. Allow approximately 24 h for stabilization and equilibration of humidity before use. Ensure that the HMK15 calibrator and the probe are at the calibration site for at least 1 h before starting the calibration in order to let the probe temperature stabilize.
- 9.3.7. Remove the capsule from the probe, exposing the sensor. Insert the sensor into a suitable hole of the LiCl salt chamber. Press it downwards till it passes through the O-rings. Wait until the humidity reading stabilizes; this will take about 10-30 min. The shorter the time the hole stays open before inserting the probe; the shorter the stabilization time required.
- 9.3.8. In the temperature range of 25-30°C, LiCl humidity changes only very slightly; thus, it is not necessary to use the thermometer. However, it may still be used to ensure that the sleeve is in the correct place. Read the salt chamber temperature from the thermometer; then read the closest humidity value from Greenspan's calibration table (Table 2).

Table 2. Greenspan's calibration table (% RH ± uncertainty).

°C	LiCl	MgCl ₂	NaCl	K ₂ SO ₄
0	*	33.7 ± 0.3	75.5 ± 0.3	98.8 ± 1.1
5	*	33.6 ± 0.3	75.7 ± 0.3	98.5 ± 0.9
10	*	33.5 ± 0.2	75.7 ± 0.2	98.2 ± 0.8
15	*	33.3 ± 0.2	75.6 ± 0.2	97.9 ± 0.6
20	11.3 ± 0.3	33.1 ± 0.2	75.5 ± 0.1	97.6 ± 0.5
25	11.3 ± 0.3	32.8 ± 0.2	75.3 ± 0.1	97.3 ± 0.5
30	11.3 ± 0.2	32.4 ± 0.1	75.1 ± 0.1	97.0 ± 0.4
35	11.3 ± 0.2	32.1 ± 0.1	74.9 ± 0.1	96.7 ± 0.4
40	11.2 ± 0.2	31.6 ± 0.1	74.7 ± 0.1	96.4 ± 0.4
45	11.2 ± 0.2	31.1 ± 0.1	74.5 ± 0.2	96.1 ± 0.4
50	11.1 ± 0.2	30.5 ± 0.1	74.4 ± 0.2	95.8 ± 0.5

- 9.3.9. Repeat Step 9.3.8 for the wet reference standard.
 - 9.3.9.1. Use NaCl as the wet reference standard (75% RH) if measured RH values are expected to be $\leq 80\%$.
 - 9.3.9.2. Use K₂SO₄ as the wet reference standard (97% RH) if measured RH values are expected to be very high (90-100%). The risk for errors increases in high humidity, and the stabilization time should be longer (about 20-40 min).
- 9.3.10. Replace the capsule.
- 9.3.11. Make necessary adjustments to the sensor output using the TXConfig program (Step 9.6)
- 9.4. Spot-checking of RH/T using the HMI41 hand-held meter or equivalent. In addition to using the salt chambers, the transmitter can be spot-checked easily by using a transfer standard. The transfer standard may be either a NIST-traceable hand-held instrument (i.e. HMI41 hand-held meter with HMP46 probe) or a fundamental measurement device such as a psychrometer. A method using the HMI41 hand-held meter is as follows:
 - 9.4.1. Ensure that the probe is correctly installed, and make sure batteries are good.
 - 9.4.2. Turn on the power. The RH/T readings should appear automatically.
 - 9.4.3. Press "MODE" to display the dewpoint temperature (Td).
 - 9.4.4. Press "HOLD" to freeze the display of the current reading. Then press "MODE" or "ENTER" to return to the normal mode.
 - 9.4.5. Press "HOLD" under the HOLD-mode, then the minimum reading after power on will show.
 - 9.4.6. Press "HOLD" under the MIN-mode to show the maximum reading. Then press any button except ON/OFF to return to the normal mode.
- 9.5. When diagnosing transmitter malfunctions, replace the sensor to check if there is a problem with the sensor, the transmitter, or both. The sensor probes are interchangeable.
 - 9.5.1. If there is dust build-up around the sensor, try using a fine brush (i.e. a paint brush) to remove the dust particles. If the dust sticks to the sensor, try dampening the paint brush slightly. Ensure the sensor has dried before taking measurements again.
- 9.6. Transmitter configuration using TXConfig
 - 9.6.1. The transmitters come pre-configured to give a 0-10 VDC analog output for 0-100% RH and 0 to 100°C. These measuring ranges can be altered, or a zero-correction factor can be added using the TXConfig software and interfacing cable set.
 - 9.6.1.1. Download the TXConfig software to the hard drive of the site computer.
 - 9.6.1.2. Connect the female DB9 connector of the TXConfig Interfacing cable to the serial port of the computer.
 - 9.6.1.3. Connect the other end of the TXConfig Interfacing cable to the transmitter using the connections shown in Fig. 3a when a power supply is used, or Fig. 3b when a 9VDC battery is used.
 - 9.6.1.4. Run TXConfig.exe.
 - 9.6.1.4.1. The TXConfig main software prompt (Fig. 4) will appear. Select the tab for RHT or RHT-WM/DM (depending on the version of software).
 - 9.6.1.4.2. Click "Read Device". The program will communicate with the sensor and display the current settings.
 - 9.6.1.4.3. The measuring ranges for RH and temperature can be adjusted, within the limits shown on the software prompt.

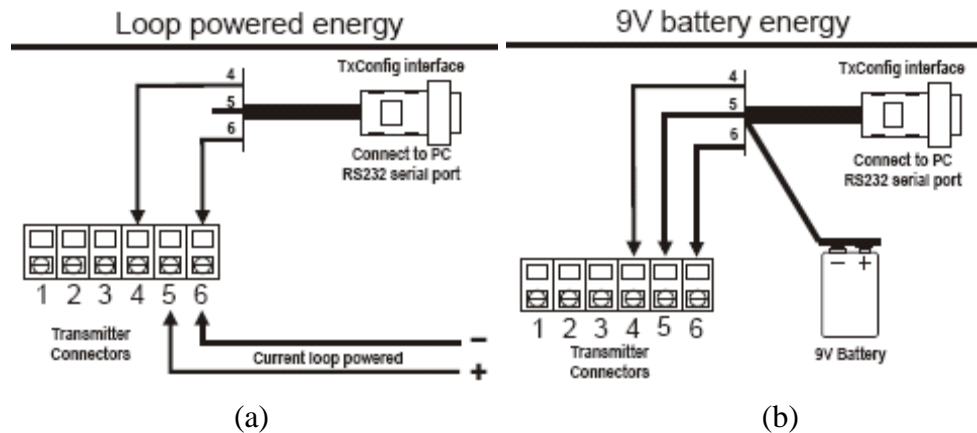


Figure 3. TXConfig Interface connections with a power supply (a) or 9VDC battery (b) power supply for sensor (Novus RHT Operating Manual)

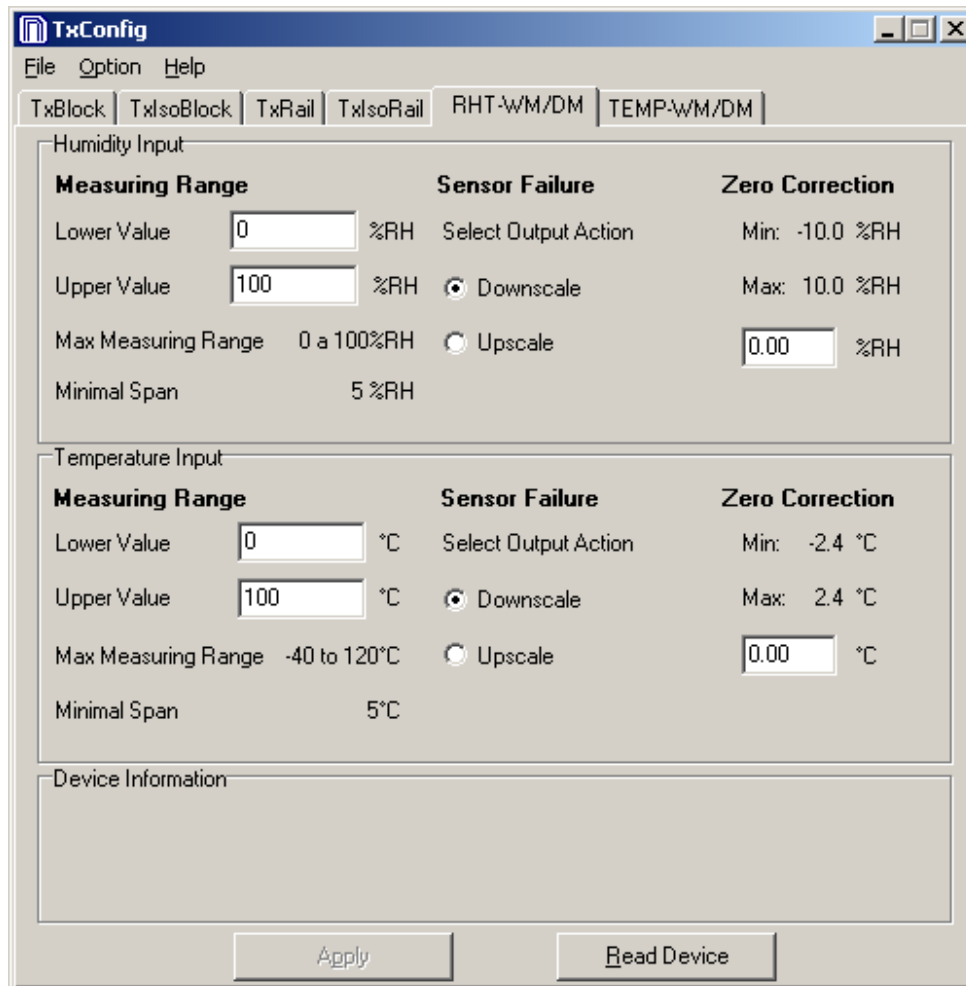


Figure 4. TXConfig software prompt form.

- 9.6.1.4.4. Click “Apply” and allow time for the computer to communicate these new settings to the sensor.
- 9.6.1.4.5. Click File – Exit to quit the TXConfig program. The cable set can be removed and the sensor can be re-installed for monitoring purposes.
- 9.7. Data Acquisition, calculations & data reduction requirements
 - 9.7.1. A 0-10 VDC analog output (0-100% RH, 0 to 100°C by default) is monitored by AirDAC.
 - 9.7.2. Report this data with 3 significant digits.
 - 9.7.3. Follow SOP B4 to correct gas concentrations for calibration and air density.
- 9.8. Computer hardware & software
 - 9.8.1. AirDAC (SOP B2)
 - 9.8.2. ADPP (SOP B3)
 - 9.8.3. CAPECAB (SOP B6)

10. Data and Records Management

- 10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
- 10.2. Manage all data according to SOP B5.
- 10.3. Document all data and information on field data sheets located in the logbook with permanent ink, and in the electronic field notes.
- 10.4. Strike out all errors with a single line. Initial and date all such corrections.

11. Quality Control and Quality Assurance

- 11.1. Calibration
 - 11.1.1. Calibration of each instrument and calibration check will performed within intervals specified in the Quality Assurance Project Plan (QAPP).
 - 11.1.2. Replace (prepare new) standard salt solutions every 6-12 mos.
 - 11.1.3. Visually check each salt solution every 2-3 mos. Discard the solution and prepare a new one if there is not at least 10% of undissolved salt at the bottom of the chamber, or the salt is not clean. LiCl may crystallize on the surface, but may still have solution under the surface. In such case, stir solution and check the next day. (If the salt solution is not going to be reused within 1 mo of its initial use, it is recommended that it should be disposed of after its use.)
 - 11.1.4. Check the O-rings on the salt chambers at each salt replacement. If they are damaged, replace with new ones. For correct calibration, it is essential that salt chambers are tightly closed.
 - 11.1.5. The uncertainties achieved using the standard salt solutions at 20°C are shown in Table 3.
- 11.2. Refer to Lemay et al. (2001) for more precautions and procedures for using RH sensors in livestock buildings.

Table 3. Uncertainties achieved using standard salt solutions at 20°C.

Salt	RH (%)	Total uncertainties (%RH)
Lithium chloride (LiCl)	11	±1.3
Magnesium chloride (MgCl ₂)	33	±1.2
Sodium chloride (NaCl)	75	±1.5
Potassium sulphate (K ₂ SO ₄)	97	±2.0

12. References

- 12.1. Novus RHT Operating Manual. 2007. Humidity and temperature transmitter operating manual. Novus, Brazil.
- 12.2. Novus RHT Datasheet. 2007. Relative humidity, dew point and temperature – RHT WM-DM. OMNI Instruments, Arroyo Grande, CA.
- 12.3. Lemay, S.P., Guo, H., Barber, E.M., and Zyla, L. 2001. A procedure to evaluate humidity sensor performance under livestock housing conditions. Canadian Biosystems Engineering 43(5): 13-21.
- 12.4. SOP B1. 2006. Data Acquisition and Control Hardware. Standard Operating Procedure B1. Purdue Ag Air Quality Lab.
- 12.5. SOP B2. 2006. Data Acquisition and Control Software (AirDAC). Standard Operating Procedure B2. Purdue Ag Air Quality Lab.
- 12.6. SOP B3. 2006. Air Data Pre-Processing Software. Standard Operating Procedure B3. Purdue Ag Air Quality Lab.
- 12.7. SOP B4. 2006. Calculation and Reporting of Air Emissions from Barns. Standard Operating Procedure B4. Purdue Ag Air Quality Lab.
- 12.8. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 12.9. SOP B6. 2006. Data Processing Software (CAPECAB). Standard Operating Procedure B6. Purdue Ag Air Quality Lab.
- 12.10. SOP P1. 2006. Measurement of Ambient Atmospheric Particulate Matter (PM₁₀) Using the Rupprecht and Patashnick TEOM Series 1400A Monitor. Standard Operating Procedure P1. Purdue Ag Air Quality Lab.

**THE MEASUREMENT OF SOLAR RADIATION WITH
THE LICOR MODEL 200SL OR 200SZ SILICON PYRANOMETER
Standard Operating Procedure (SOP) E3**

**THE MEASUREMENT OF SOLAR RADIATION WITH
THE LICOR MODEL 200SL OR 200SZ SILICON PYRANOMETER
Standard Operating Procedure (SOP) E3**

Prepared by

Juan Carlos Ramirez, Bill W. Bogan, and Richard H. Grant

Reviewed by

Albert J. Heber, Ji-Qin Ni, and Kenneth D. Casey (Texas A&M Univ.)

Effective Date: November 6, 2007

**PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)
Agricultural and Biological Engineering, Purdue University
West Lafayette, IN 47907**

Table of Contents

	Page
1. Scope and Applicability.....	3
2. Summary of Method.....	3
3. Definitions.....	4
4. Health and Safety.....	4
5. Cautions.....	4
6. Interferences.....	4
7. Personnel Qualifications.....	5
8. Equipment and Supplies.....	5
9. Procedures.....	5
10. Data and Records Management.....	8
11. Quality Control and Quality Assurance.....	8
12. References.....	9
13. Contact Information.....	9
Appendix A. Calibration form: LI-200SL Solar Radiation Sensor.....	10
Appendix B. Procedure to Calculate Potential Solar Radiation on a Horizontal Surface....	11

1. Scope and Applicability

- 1.1. The method described in this SOP is to use the silicon photodiode to measure solar incident radiation in the 300 to 1200 nm wavelength band.
- 1.2. This procedure applies only to the LiCOR Model LI-200SL Pyranometer for monitoring solar radiation during barn air emissions measurements, or for atmospheric stability class determination. By default, these instructions apply to both measurement scenarios. If there are any differences between the two applications, this will be specified.
- 1.3. Detection limit is 10 W/m^2 . Ambient range is 10 to 1300 W/m^2 .

2. Summary of Method

A pyranometer is an instrument for measuring solar radiation. The Model LI-200SL Pyranometer (LiCOR, Lincoln, NE) includes a Model LI-200SA pyranometer sensor and a millivolt converter. The sensor is installed on a mounting and leveling fixture to receive solar radiation (Figure 1). It uses a silicon-based photodiode semiconductor that converts the solar energy into a microamp current proportional to the solar radiation. The current signal is provided to a converter through a BNC connector. The converter contains a variable resistor that is unique to the LI-200SA sensor, and that converts the current signal to a standardized output voltage, 10 mV per 1000 W/m^2 . The ultimate termination of the converter consists of bare leads to be connected to the data acquisition system, which converts the voltage signal to solar radiation in W/m^2 , displays it on a computer monitor, and saves it into data files.

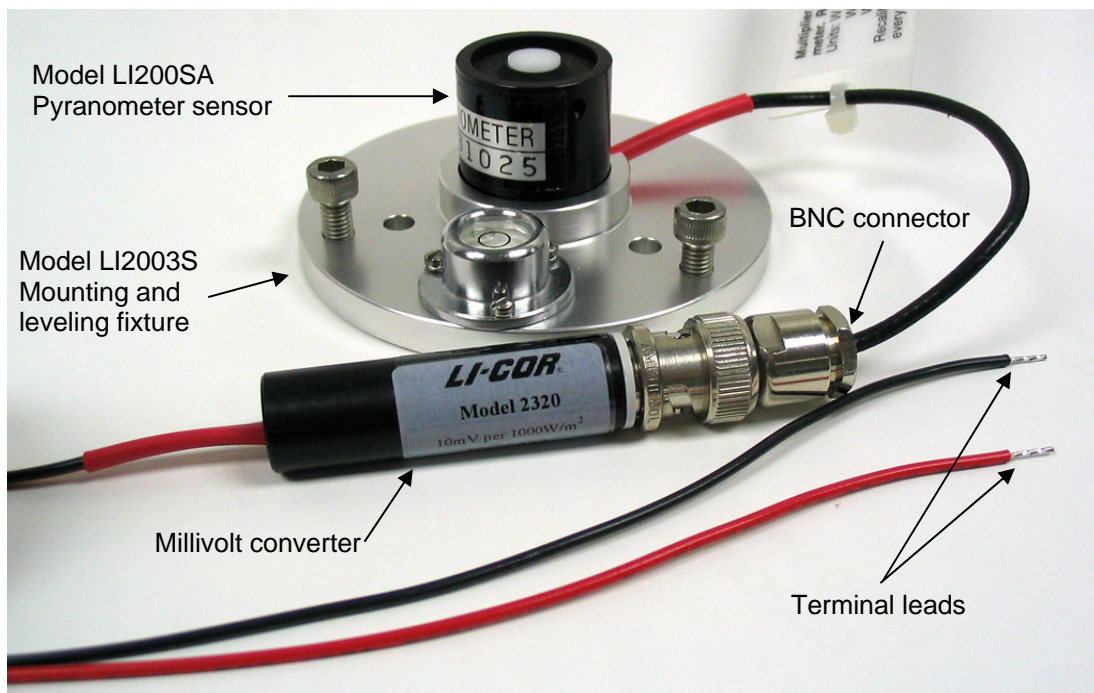


Figure 1. Model LI-200SL Pyranometer (including a LI-200SA Pyranometer sensor and a LI-2302 standardized millivolt adapter) and Model LI2003S Mounting and Leveling Fixture (adapted from LI-COR, 2006a).

3. Definitions

- 3.1. BNC Bayonet Neill Concelman connector (used with coaxial cables)
- 3.2. PAAQL Purdue Agricultural Air Quality Laboratory
- 3.3. QAPP Quality Assurance Project Plan

4. Health and Safety

- 4.1. The pyranometer only produces weak electric signal that does not pose a health concern.
- 4.2. The pyranometer is installed outdoors, usually several meters above the ground. Follow all relevant safety precautions during installation and maintenance. Locate the sensor where it can be reached safely by the operator during the periodic clean up procedures.

5. Cautions

- 5.1. The acrylic material used in LI-COR light sensors can be crazed by exposure to alcohol or organic solvents, which adversely affects the response of the sensor. Clean the diffuser element of the sensor only with water and mild soap such as dishwashing soap. Do not use alcohol, organic solvents, abrasives, or strong detergents. LI-COR has found that vinegar can also be used to remove hard water deposits from the diffuser element, if necessary.
- 5.2. The spectral response of the LI-200 does not include the entire solar spectrum. Therefore, use the pyranometer in the same lighting conditions as those under which it was calibrated. Use it only to measure unobstructed daylight, not reflected sunlight.
- 5.3. Remove and save the red protective cap from the sensor. This is used to protect the sensor when it needs to be calibrated or stored.
- 5.4. Be careful when connecting the sensor cable to the data acquisition system. Make sure the connections are tight, and are made according to SOP B1.
- 5.5. Each pyranometer sensor is used only with the millivolt converter that is supplied with it and the serial numbers must match in the Model LI200SL. Be sure to change both the sensor and the converter if the LI200SL needs to be replaced.
- 5.6. The millivolt converter is not weatherproof. Install it inside the instrument shelter or protect it in a weatherproof enclosure.

6. Interferences

- 6.1. Incorrect siting of the solar radiation sensors can lead to inaccuracies over the period of data collection. One example is installing the sensor where it receives the shadow of a building or other object. Another example is installing the sensor where it receives the reflection of a clear painted wall or other object. It should not be used under vegetation.
- 6.2. Particulate matter and bird droppings on the optical head will cause low readings. The risk of this interference is greater near livestock buildings.
- 6.3. The signal provided by the converter to the data acquisition is a weak voltage. To prevent excess signal noise pickup, avoid using long cable between the converter and the data acquisition system or running the cable close to power cords.

7. Personnel Qualifications

- 7.1. Personnel must be trained in the use of the handheld millivolt multimeter before initiating the procedure. Training requires 10 minutes.
- 7.2. Each member of the field staff must read the pertinent sections of the sensor instruction manual (pp 1-5, 10-13) and this SOP before operating this instrument.

8. Equipment and Supplies

- 8.1. For barn measurement locations:
 - 8.1.1. Model LI-200SL Pyranometer (LiCOR, Inc., Lincoln, NE), which includes a LI-200 SA pyranometer, a BNC connector and a millivolt adapter. The LI-200SL comes with a 15-ft cable.
- 8.2. For open source measurement locations:
 - 8.2.1. Model LI-200SZ Pyranometer (no BNC connector) with Model 2220 millivolt adaptor (LiCOR, Inc., Lincoln, NE)
 - 8.2.2. Weatherproofed NEMA 4X box
 - 8.2.3. Data logger (SOP W6)
- 8.3. Model LI2003S Mounting and Leveling Fixture (LiCOR, Inc.)
- 8.4. Sensor stand and mounting arm (Fig. 2) if they are not available in the weather tower.
- 8.5. Machine screws with nuts to mount the Mounting and Leveling Fixture to the sensor stand.
- 8.6. 3/64" hex wrench to mount the pyranometer sensor to the Mounting and Leveling Fixture, and 1/8" screwdriver to fasten the screws
- 8.7. Plastic cable ties to fix the sensor cable
- 8.8. Multimeter to check the pyranometer signal output, with a resolution of ≤ 0.01 mV
 - 8.8.1. Multimeter must be pre-calibrated for open-source applications. This is not necessary for barn applications.
- 8.9. Soft cloth, soap, water and soft toothbrush if the sensor needs cleaning
- 8.10. Electronic parts cleaner
- 8.11. Heat shrink tubing
- 8.12. Heat blower
- 8.13. Unused LiCOR LI-200 (to calibrate the sensor)

9. Procedures

- 9.1. Acceptance
 - 9.1.1. Verify that the serial number on the calibration certificate matches the serial number on the instrument.
 - 9.1.2. Verify that the serial numbers of the pyranometer sensor and the converter match.
 - 9.1.3. Response check:

- 9.1.3.1. Place pyranometer outside facing towards the sky on a sunny day.
- 9.1.3.2. Remove and save the red protective cap from the sensor.
- 9.1.3.3. Connect the sensor and the converter with the BNC connector.
- 9.1.3.4. Measure sensor response across the positive and negative wires with a multimeter.
 - 9.1.3.4.1. The multimeter resolution should be better than 0.01 VDC. For atmospheric stability classification uses, the multimeter should be a calibrated model.
- 9.1.3.5. If sensor voltage is less than 1 mV, return sensor to supplier for replacement.
- 9.1.4. Zero check:
 - 9.1.4.1. Obstruct all light from entering the optical window using a non-reflective cup or other means and measure the voltage with a millivolt multimeter across the sensor wires.
 - 9.1.4.2. Value should be less than 0.01 mV. If value is greater than 0.01 mV, return the instrument to supplier for replacement.
- 9.1.5. Installation
- 9.1.6. Mount the Mounting and Leveling Fixture to the horizontal platform, 2 ft away from the S side of the Weather Station Tower (or the S side of a 10-ft mast, in open-path measurements) using a mounting arm or a cross arm stand (Fig. 2), such that no obstruction can cast a shadow over the sensor, according to SOP E5 (barn installation), SOP U5 (lagoon/basin installation), or U7 (corral installation).

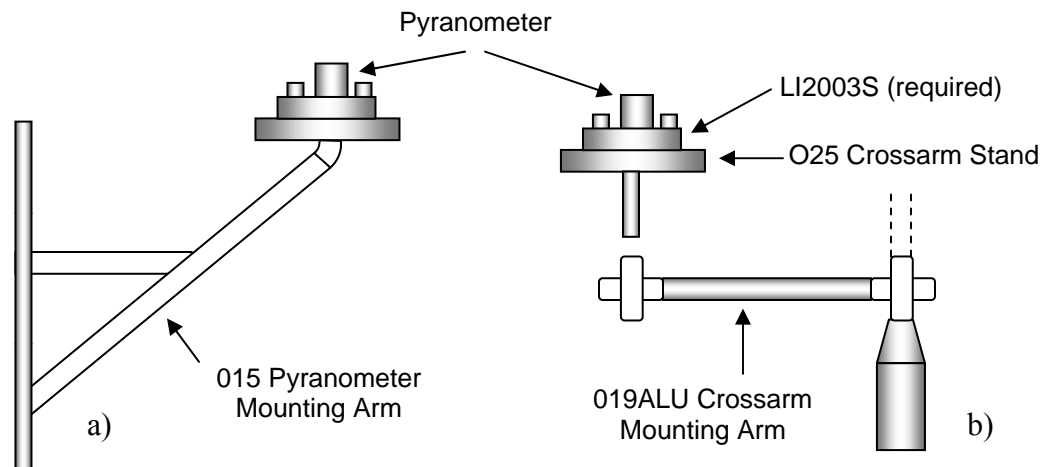


Figure 2. Pyranometer mounting arm (a) and cross arm stand (b) (LiCor Inc., 2006b).

- 9.1.7. Securely mount the pyranometer sensor onto the Mounting and Leveling Fixture using the 3/64" hex wrench.
- 9.1.8. Adjust the level of the Mounting and Leveling Fixture.
- 9.1.9. If installing the sensor for open-source use, pass the coaxial cable through the appropriate knockout of the weatherproofed NEMA 4X box.

- 9.1.10. If installing the sensor for barn use, no NEMA 4X box is needed. Wrap the BNC connector, and its junction with the millivolt adapter, with electrical tape.
- 9.1.11. Run the cable from the pyranometer to the instrument shelter or trailer.
 - 9.1.11.1. If a field datalogger is being used (i.e. during open-source applications), use the 1/8" slotted screwdriver to connect the positive signal wire to the single-ended measurement channel, and the black wire to ground according to SOP W6.
- 9.1.12. Connect the cable to the Millivolt Converter inside the instrument shelter.
- 9.1.13. Measure voltage across the two wires using a multimeter. On a sunny day, the voltage should be at least 1 mV.
- 9.1.14. Attach wires to data acquisition system according to SOP B1.
- 9.2. Calibration checks
 - 9.2.1. Check the calibration of a pyranometer which is in use by comparing its readings with an unused pyranometer of the same model which is known to be accurate.
 - 9.2.1.1. This can be done in the field or at Purdue. The QAPP for a particular project will state which option is to be used.
 - 9.2.1.1.1. If these checks are to be done in the field, it is necessary to include an additional support and additional wiring for the collocated sensor, as well as the corresponding wiring, when installing the sensor. There must also be an available input channel in the data acquisition system for this collocated sensor.
 - 9.2.1.1.2. This is not necessary if the instruments are to be sent to Purdue for calibration.
 - 9.2.1.2. Measurements are made every 5 min for 2 d, using the same data acquisition system used in the study to collect data from both sensors.
 - 9.2.1.3. The calibration check frequency will be specified in the QAPP.
 - 9.2.1.3.1. The manufacturer recommends recalibrating this instrument every two years. A shorter interval can be chosen based on the QAPP's Quality Objectives.
 - 9.2.2. Calculate the difference in each 5-min paired measurement, and compute the mean difference between the two sensors for the daylight hours over the two days.
 - 9.2.3. If the mean difference between the sensor under calibration and the unused sensor is $< \pm 5\%$ of the unused sensor's reading, then the sensor being calibrated is assumed good. In this case, calculate a correction factor to apply to the in-field sensor.
 - 9.2.4. If the mean difference between the sensor under calibration and the unused sensor is greater than $\pm 5\%$ of the unused sensor's reading, then check the cleanliness and the connections of the sensor that is being checked, and repeat the calibration check. If the difference persists, the sensor being checked is assumed to be defective, and must be returned to the supplier for repair or replacement.
- 9.3. Troubleshooting
 - 9.3.1. Low readings are often due to a dirty optical window. Clean surface of sensor with soap and water.
 - 9.3.2. Erratic values are often due to poor connections. Check connections for tightness.
- 9.4. Data acquisition, calculations & data reduction requirements
 - 9.4.1. The LI-200SL output is acquired by a data acquisition (DAQ) system (SOP B1 and SOP B2) at a frequency of 1 Hz.

- 9.4.2. The DAQ system converts the voltage signals to W/m^2 , averages them every 15 s and 60 s, and saves the averaged data in data files.

10. Data and Records Management

- 10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method, as well as in a bound record book designated for the method.
- 10.2. Manage all data according to SOP B5 (for barn measurements) SOP D1 (for open-source meteorological measurements).
- 10.3. Document all data and information on field data sheets and in site logbooks with permanent ink, or in electronic notes.
- 10.4. Strike out errors in writing with a single line. Initial and date all such corrections.

11. Quality Control and Quality Assurance

- 11.1. Do the following at the beginning and end of each measurement period, or as specified in the QAPP:
 - 11.1.1. Check physical cleanliness of the pyranometer's optical window. If dirty, clean with soap and a wet cloth. Always use lens cloth to avoid scratching the glass surface.
 - 11.1.2. Perform recalibration if the optical window shows any scratches, or discoloration that cannot be washed off.
 - 11.1.3. Check physical cleanliness of connectors. If corroded, clean with electronic cleaner and toothbrush.
 - 11.1.4. Check integrity of the plastic casing of the signal cable.
 - 11.1.4.1. If damaged slightly, repair with heat-shrink tubing.
 - 11.1.4.2. If damaged significantly, replace sensor and return sensor to manufacturer for repair.
 - 11.1.5. Perform zero checks (Section 9.1.4).
 - 11.1.6. Record results of each check in the calibration file (Appendix A) for open-source applications, or a similar table entry in the electronic field notes for barn applications.
 - 11.1.7. Calibration checks against a collocated sensor (Section 9.2).
- 11.2. Do the following daily:
 - 11.2.1. Compare the measurement with potential irradiance based on time of day, latitude and longitude of the location, and a solar constant of $1367 W/m^2$ (Appendix B).
 - 11.2.2. Ratio of sensor measurement to potential irradiance must be less than 0.9.
 - 11.2.3. If ratio is greater than 0.9:
 - 11.2.3.1. Flag data value as invalid.
 - 11.2.3.2. Perform a calibration check against an unused pyranometer (Section 9.2). If the sensor fails this check (Section 9.3.4), replace sensor and return sensor to factory for repair.
 - 11.2.4. Check measurements between 10 AM and 2 PM.
 - 11.2.4.1. If measurements are less than $100 W/m^2$:
 - 11.2.4.1.1. Flag data values for day as invalid
 - 11.2.4.1.2. Check wiring and optical window. Repair, clean, or replace as necessary.

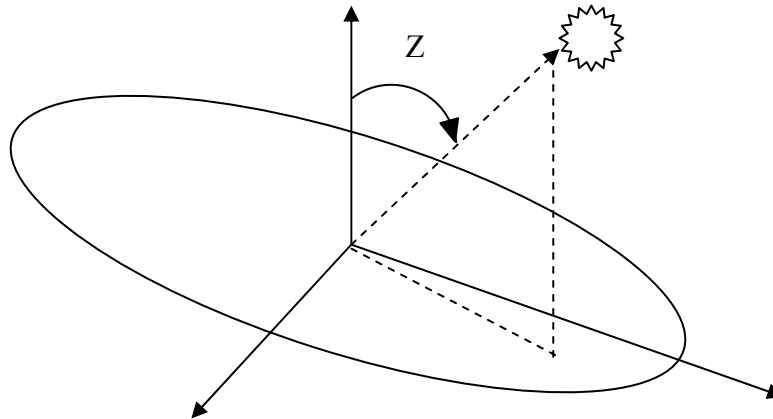
12.

12. References

- 12.1. LI-COR. 1986. LI-COR Radiation Sensors Instruction Manual: Terrestrial Type SA. Publication No. 8609-56. Revised July, 1991. Available at http://ftp.licor.com/env/Radiation_Sensors/Manual/Terrestrial_SA_Manual.pdf. Accessed on 2/2/06.
- 12.2. LI-COR, Inc. 2006a. Pyranometer LI-200SL. Available at <http://www.licor.com/env/Products/Sensors/rad.jsp> Accessed on 2/3/2006.
- 12.3. LI-COR, Inc. 2006b. Pyranometer LI-200X. Available at <http://www.campbellsci.com/documents/manuals/li200x.pdf>. Accessed on 2/3/2006.
- 12.4. USEPA. 1995. Quality Assurance Handbook for Air Pollution Measurement Systems, Volume IV- Meteorological Measurements. Document No. EPA/600/R-94/038d.
- 12.5. USEPA. 2000. Meteorological Monitoring Guidance for Regulatory Modeling Applications, EPA-454/R-99-005.
- 12.6. NCDAQ. 1994. General Guidelines for On-Site Meteorological Data Collection for N.C. Air Quality Analysis.
- 12.7. SOP B1. 2006. Data Acquisition and Control Hardware. Standard Operating Procedure B1. Purdue Ag Air Quality Lab.
- 12.8. SOP B2. 2006. Data Acquisition and Control Software (AirDAC). Standard Operating Procedure B2. Purdue Ag Air Quality Lab.
- 12.9. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 12.10. SOP D1. 2006. Management of Open-source, Weather, and Lagoon-characterization Data. Standard Operating Procedure D1. Purdue Ag Air Quality Lab/ Purdue Applied Meteorology Lab.
- 12.11. SOP E5. 2006. Roof-mounted Weather Station Tower. Standard Operating Procedure E5. Purdue Ag Air Quality Lab.
- 12.12. SOP U5. 2006. The Installation of Open-source Measurement Equipment. Standard Operating Procedure U5. Purdue Ag Air Quality Lab/ Purdue Applied Meteorology Lab.
- 12.13. SOP U7. 2006. The Instrument Trailer and Installation of Open-Source Measurement Equipment (Corrals). Standard Operating Procedure U7. Purdue Ag Air Quality Lab/ Purdue Applied Meteorology Lab.
- 12.14. SOP W6. 2006. Establishment, Data Acquisition and Control of Weather and Lagoon Characterization Hardware. Standard Operating Procedure W6. Purdue Ag Air Quality Lab/ Purdue Applied Meteorology Lab.
- 12.15. National Oceanic and Atmospheric Administration (NOAA). Solar Position Calculator. Available on <http://www.srrb.noaa.gov/highlights/sunrise/azel.html>. Accessed on 7/10/2006.
- 12.16. Free Zip Code Lookup. Available at <http://zipinfo.com/search/zipcode.htm>. Accessed on 7/10/2006

13. Contact Information

- 13.1. Distributor: Campbell Scientific, Inc., 815 West 1800 North, Logan, Utah, 84321. Technical support (435)-753-2342.

Appendix B. Procedure to Calculate the Potential Solar Radiation on a Horizontal Surface.

Calculate the vertical component of the potential solar radiation on a horizontal surface using the following equation:

$$H_o = I_o * \text{Cos}Z$$

Where:

H_o = Potential extraterrestrial insolation on a horizontal surface (W/m^2)

I_o = Extraterrestrial solar radiative flux ($1367 \text{ W}/\text{m}^2$)

Z = zenith angle (from the vertical position to the position of the sun).

Obtain the site-specific Cosine of the Zenith angle ($\text{Cos}Z$) from the National Oceanic and Atmospheric administration (NOAA) website (<http://www.srrb.noaa.gov/highlights/sunrise/azel.html>), by entering the geographic location of the monitoring site (latitude and longitude) and the time of day. The online calculator will determine the cosine of the zenith.

If unsure of the latitude and longitude of the specific monitoring site, this can be obtained through the website <http://zipinfo.com/search/zipcode.htm>, by checking the “latitude and longitude” box and the entering the ZIP code of the location.

**WIND SPEED AND DIRECTION MEASUREMENTS
USING THE R. M. YOUNG MODEL 03002VM WIND SENTRY
Standard Operating Procedure (SOP) E4**

**WIND SPEED AND DIRECTION MEASUREMENTS
USING THE R. M. YOUNG MODEL 03002VM WIND SENTRY
Standard Operating Procedure (SOP) E4**

Prepared by

Juan Carlos Ramirez

Reviewed by

**Ji-Qin Ni, Albert Heber, Bill W. Bogan,
and Kenneth D. Casey (Texas A&M Univ.)**

Effective Date: November 7, 2007

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability.....	3
2. Summary of Method.....	3
3. Definitions.....	4
4. Health and Safety.....	4
5. Cautions.....	4
6. Interferences.....	4
7. Personnel Qualifications.....	5
8. Equipment and Supplies.....	5
9. Procedure.....	5
10. Data and Records Management.....	10
11. Quality Control and Quality Assurance.....	10
12. References.....	11
13. Supplier Contact Information.....	11

1. Scope and Applicability

- 1.1. Wind speed and direction data is needed to provide local weather conditions, which can help explain some abnormalities registered by other instruments.
 - 1.1.1. One important example of this is the effect of wind on the performance of the barn's low-pressure exhaust fans.
- 1.2. This SOP describes the installation, operation and maintenance of the R.M. Young Model 03002VM Wind Sentry. The range of this instrument is 0-50 m/s (0-112 mph).
 - 1.2.1. This method has a wind speed threshold of 1.1 m/s (2.5 mph).
- 1.3. This instrument is typically installed on top of a tower at barn emission sites, on or near the barn where wind speed and direction are secondary variables.

2. Summary of Method

The R.M. Young Model 03002VM Wind Sentry (Fig. 1) measures horizontal wind speed and direction. Cup rotation produces an AC sine wave voltage signal with a frequency proportional to wind speed. The vane orientation is sensed by a 10 k Ω precision conductive plastic potentiometer. Internal circuitry converts both of these signals to a linear 0-5 V voltage signal. Signal conditioning electronics are integrated into the instrument junction box. The mounting ring is secured to the mounting pipe after the proper vane orientation is verified.

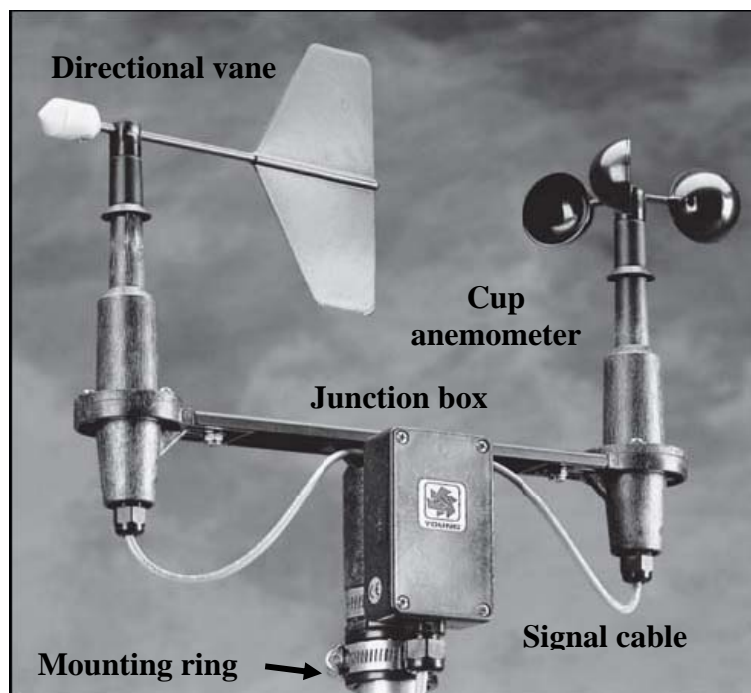


Figure 1. R.M. Young Model 03002VM Wind Sentry (R.M. Young Company).

3. Definitions

- 3.1. DAS Data acquisition system
- 3.2. AirDAC Data acquisition and control software (See SOP B2)
- 3.3. PAAQL Purdue Agricultural Air Quality Laboratory
- 3.4. QAPP Quality Assurance Project Plan

4. Health and Safety

- 4.1. Any work performed on the roof when installing weather monitoring equipment, including the wind monitor, should follow the OSHA (U.S. Occupational Safety and Health Administration) requirements for roof work and ladder safety, as covered in Section 4 of SOP E5.
- 4.2. Use a safety harness when climbing or working on a tower.
- 4.3. Towers should preferably be lowered to the ground to install the wind monitor.

5. Cautions

- 5.1. Grounding the Wind Sentry is vitally important. Without proper grounding, static electrical charge can build up during certain atmospheric conditions and discharge through the transducers, inducing erroneous signals or transducer failure.
 - 5.1.1. Do not paint the contact surface between the instrument and the tower. The instrument is grounded using the metal pipe or tower structure. The tower must be connected to a grounding rod.
 - 5.1.2. The instrument "Earth Ground" (Fig. 3) on the circuit board assembly inside of the junction box must be connected to an earth ground using the shield of one of the signal cables that takes the wind speed signal or the wind direction signal to the data acquisition computer. Connect this shield to the same ground as the computer.
- 5.2. Bearing damage can cause the wind speed threshold value to increase. Check the bearings whenever noise is detected or the speed threshold increases.
- 5.3. The potentiometer used to measure the wind direction requires a DC excitation voltage of 8-24 V. Do not exceed 24 VDC.

6. Interferences

- 6.1. Proper grounding prevents electrostatic noise from affecting the signal.
- 6.2. Nearby obstacles (trees, tall buildings, etc) can affect wind patterns, and result in inaccurate data.
- 6.3. The building upon which a roof tower is mounted can interfere with the measurement. This interference is acceptable for secondary measurements.
- 6.4. The directional sensor has an 8° deadband region from 352° to 360°, although the mechanical range of the instrument is 360°. The signal conditioning of the 03002VM has an active "pull up circuit". This means that when the wiper of the potentiometer is in the 8° deadband area, instead of the output signal floating electrically giving random

values, the output is pulled up to full scale. The instrument cannot be used to distinguish wind directions between 352° and 360°.

7. Personnel Qualifications

- 7.1. Installer and operator must read this SOP before attempting to install or do any maintenance activity on the instrument.
- 7.2. Installer or operator must read the “Health and Safety” Section (Section 4) of SOP E5, which covers precautions for working on roofs and using ladders.
- 7.3. Installer must understand how to wire signal cables.
- 7.4. Personnel responsible for installing the wind monitor tower should be comfortable with heights.

8. Equipment and Supplies

- 8.1. Equipment
 - 8.1.1. Wind Sentry (Model 03002VM, R. M. Young Co., Traverse City, MI), with 0-5 VDC outputs for both wind speed and direction
 - 8.1.2. Shielded signal cable, 2-pair, 22 AWG
 - 8.1.3. Vane angle bench stand (Model 18112, R. M. Young Co.) to calibrate direction (optional)
 - 8.1.4. Anemometer Drive (200 to 15,000 rpm) to calibrate wind speed (Model 18802, R.M. Young Co.)
 - 8.1.5. Propeller torque disc (Model 18314, R. M. Young Co.)
 - 8.1.6. Compass
 - 8.1.7. Ladder that extends at least 3 ft above the roof or landing surface
 - 8.1.8. Safety harness system (to work on roof or tower)
 - 8.1.9. Slip-resistant shoes (to work on roof or tower)
 - 8.1.10. Plastic cable ties (to fasten signal and electric cables).
- 8.2. Critical spare parts
 - 8.2.1. The instrument parts likely to need replacement (due to normal wear) are the precision bearings and the potentiometer:
 - 8.2.1.1. Flange bearing (2 each on the cup and vane shafts) 03126S
 - 8.2.1.2. Potentiometer mounting & coil assembly 03330A

9. Procedure

- 9.1. Receiving inspection check
 - 9.1.1. Upon receipt, check the instrument for any visual shipping damage.
- 9.2. Initial performance checkout

Before installing the instrument on the roof tower, bench test the instrument mechanically and electrically.

 - 9.2.1. Initial mechanical checkout
 - 9.2.1.1. Using the mounting ring, install the instrument on a suitable support in its working position.

- 9.2.1.2. Place the cup wheel on the anemometer shaft, and tighten the set screw on the side of the hub.
- 9.2.1.3. Check that the vane and the cup wheel rotate freely without any friction.
- 9.2.1.4. Blow gently on the cups. Watch for obvious high torque or irregular rotation.
- 9.2.1.5. Check vane balance by holding the instrument base so the vane surface is horizontal. It should have near-neutral torque without any tendency to rotate. A slight imbalance will not degrade performance.
- 9.2.1.6. The anemometer bearing conditions can be checked using a propeller torque disc. This disc allows verification of the anemometer bearing torque with 0.1 g-cm resolution. The disc temporarily replaces the cup wheel for torque measurement. The disc also can be used for simple and accurate pass/fail checks. Charts included with the disc relate torque to propeller threshold with limits for acceptable bearing performance.
- 9.2.2. Initial check of outputs of wind speed and direction circuits
- 9.2.2.1. Connect excitation voltage and signal cables to terminal strip, located in the junction box (Figure) according to the wiring diagram in the instrument manual. If the instrument cannot be connected to the DAC system that will be used during data collection, a multimeter can be used.
- 9.2.2.2. Connect the “Earth Ground” terminal from the junction box (Fig. 2) to the shield of one of the two paired cables that are used to transmit the wind speed signal or the wind direction signal. Then connect this ground to the computer ground.
- 9.2.2.3. The excitation voltage must not exceed 24 VDC.

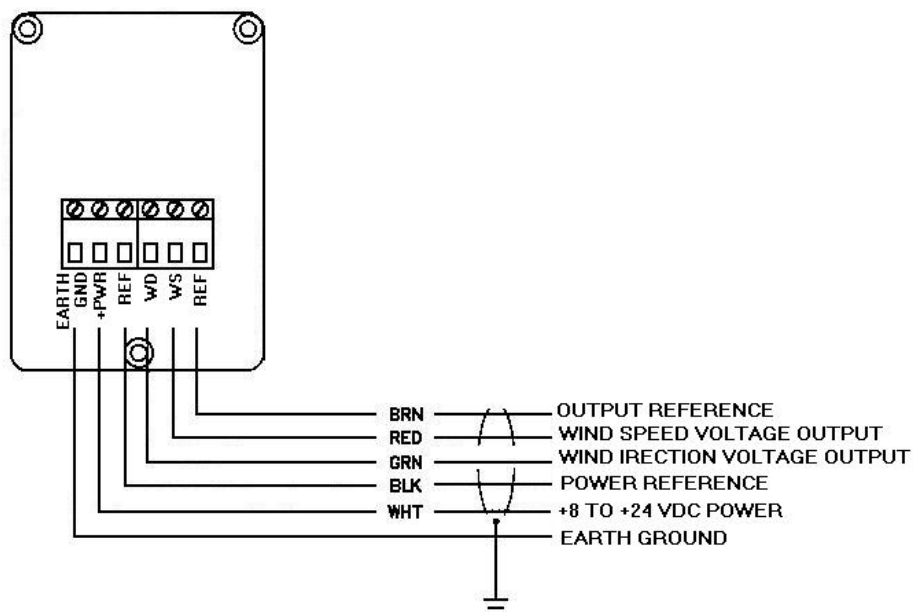


Figure 2. Wind anemometer PC board in junction box (Model 03002V Manual).

- 9.2.2.4. The output of the propeller rotation can be calibrated using an Anemometer Drive (R. M. Young Model 18802 or equivalent) attached to the anemometer shaft (after disconnecting the cup wheel). Apply the appropriate calibration formula (Section 9.4.3.2 in this SOP) to the calibrating motor rpm and adjust the electronics to the proper value.

9.3. Installation

9.3.1. Mount Wind Sentry (Fig. 3)

- 9.3.1.1. Install the mounting post on the tower (SOP E5).
 9.3.1.2. Place Wind Sentry on mounting post. Do not tighten the mounting ring band clamp yet.

9.3.2. Connect Instrument Cable

- 9.3.2.1. Follow the instructions presented in Section 9.2.2.1 of this SOP. Also, refer to the Wind Sentry cable and wiring diagram included in the instrument manual.

9.3.3. Align Vane

- 9.3.3.1. Connect the anemometer to the DAC system (which will show wind direction in angular degrees), or to a multimeter (which will measure voltage output only).
 9.3.3.2. Point the instrument in a known direction
 9.3.3.2.1. If a known directional reference point exists, this may be used.
 9.3.3.2.2. A compass may be used to orient the instrument in a known direction, if the values are considered secondary or auxiliary data.
 9.3.3.3. Sighting down the vane centerline, point the counterweight on the vane at the reference point, or in the direction indicated by the compass.
 9.3.3.4. While holding vane in position, slowly turn base until the indicator shows the proper value.
 9.3.3.4.1. If the DAC system is used as the indicator, the proper value will be the direction, expressed in angular degrees (0 = due north).
 9.3.3.4.2. If a multimeter is used, the 0-5 VDC output of the instrument must be adjusted to 0-4.89 VDC, given the 8-degree deadband (Section 6.4). The proper output is thus determined by:

$$\frac{\text{Orientation (angular degrees)}}{352^\circ} \times 4.89 \text{ VDC} \quad (1)$$

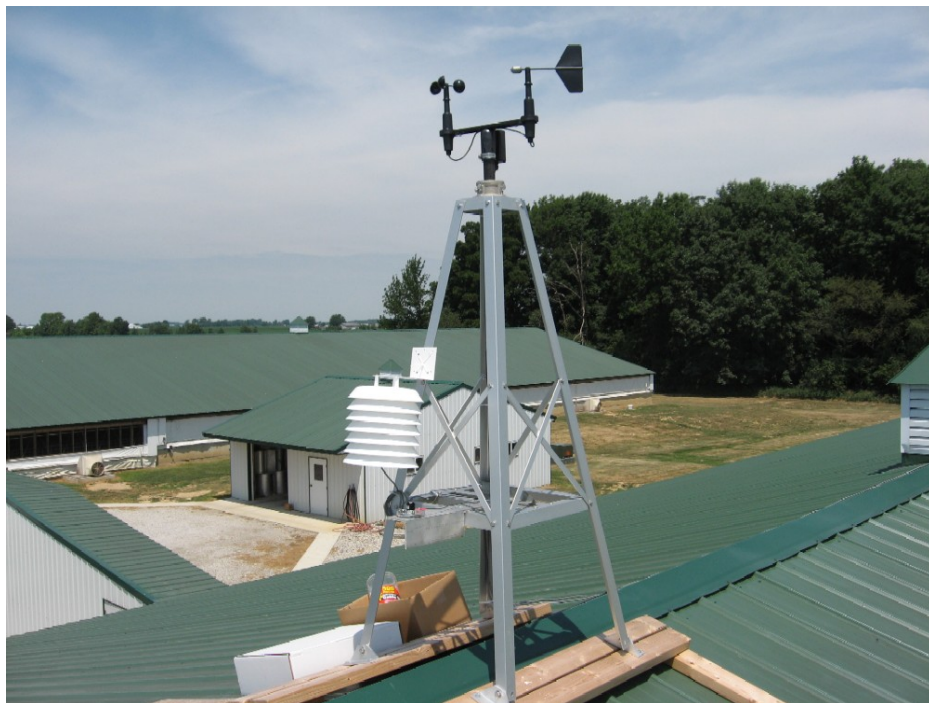
- 9.3.3.5. Tighten mounting post band clamp.

9.3.4. Grounding the instrument

- 9.3.4.1. Follow the steps described in Section 9.2.2.2.



(a)



(b)

Figure 3. Wind Sentry installation next to the on-farm instrument shelter at a site (a), and a close view of the Wind Sentry and weather station tower on the barn roof at another site (b) (PAAQL files).

9.4. Instrument calibration

- 9.4.1. The wind monitor is fully calibrated before shipment and should require no adjustments. If recalibration is necessary after maintenance, send it to the manufacturer.
- 9.4.2. Certain applications of the wind direction data require an accurate wind direction calibration, which can be performed using the vane angle bench stand. Other applications of the data require a less accurate calibration, such as secondary data situations. In this case, the calibration of the instrument can be checked using a compass. Consult the QAPP for the particular project to determine which directional calibration is needed.
- 9.4.3. Wind speed calibration is determined by the cup wheel turning factor and the output of the transducer. Standard accuracy is ± 0.3 m/s (0.6 mph) for air speed and $\pm 3^\circ$ for wind direction. For greater accuracy, the instrument must be individually calibrated in comparison with a NIST-traceable wind speed standard.
- 9.4.3.1. To calibrate Wind Sentry electronics using a signal from the instrument, temporarily remove the cup wheel and connect the anemometer drive to the cup wheel shaft. Apply appropriate calibration formula to the calibrating motor rpm, and adjust the electronics for the proper value. The calibration formula relates wind speed to cup wheel rpm. This calibration formula was obtained by the manufacturer after wind tunnel tests on the specific cup wheel design.
- 9.4.3.2. Calibration formulas for the wind monitor with propeller:
- 9.4.3.2.1. Wind speed vs. cup wheel rpm
- $$\begin{aligned} \text{m/s} &= (0.01250 \times \text{rpm}) + 0.2 \\ \text{knots} &= (0.02427 \times \text{rpm}) + 0.4 \\ \text{mph} &= (0.02795 \times \text{rpm}) + 0.4 \\ \text{km/h} &= (0.04499 \times \text{rpm}) + 0.7 \end{aligned}$$
- 9.4.3.2.2. Wind speed vs. output voltage (0-5 V)
- $$\begin{aligned} \text{m/s} &= 0.25 \times \text{mV} \\ \text{knots} &= 0.50 \times \text{mV} \\ \text{mph} &= 0.50 \times \text{mV} \\ \text{km/h} &= 1.00 \times \text{mV} \end{aligned}$$
- 9.4.3.2.3. For example, to determine indicated wind speed (in m/s) when cup wheel is turning at 1000 rpm, perform the following calculations:
 $(1000 \text{ rpm} * 0.01250 \text{ (m/s)/rpm}) + 0.2 = 12.7 \text{ m/s}$.
12.7 m/s should be the value indicated by the display of the DAC system.
If not, adjust the DAC system as required.
- 9.4.3.3. Torque verification (bearing/transducer condition verification)
- 9.4.3.3.1. Remove the Wind Sentry from the tower, remove the cup wheel from the anemometer shaft, and replace with the torque disc.
- 9.4.3.3.2. Use torque disc to measure anemometer torque in both directions (clockwise and counterclockwise). Using a combination of black nylon screws (0.1 g) and pan-head stainless steel screws (1.0 g), place weights at the various radii on the torque disc to create the desired torque. Hold torque disc with the weights in a horizontal line, then release the disc. Disc should rotate freely downwards.

- 9.4.3.3.3. For a new anemometer, the acceptable threshold is 1.2 g-cm. This corresponds to a stainless steel screw at the 1-cm radius, plus a nylon screw at the 2-cm radius. For a used anemometer, in order to maintain a threshold of 1.1 m/s, this same maximum acceptable torque value applies.
 - 9.4.3.3.4. Failure to freely rotate downwards indicates degraded bearings, which require service.
- 9.5. Troubleshooting
- 9.5.1. If noise from the Wind Sentry is detected:
 - 9.5.1.1. Check grounding of the instrument.
 - 9.5.1.2. Check flange bearings on the top and bottom of the cup wheel shaft or vane shaft (as appropriate, depending on which signal(s) is noisy).
 - 9.5.2. If wind speed threshold increases, check flange bearings on the cup wheel shaft.
 - 9.5.3. If signal output is nonlinear, check the potentiometer and/or replace it.
- 9.6. Data acquisition, calculations & data reduction requirements
- 9.6.1. The Wind Sentry signals (velocity and direction) are acquired by the data acquisition system according to SOP B2.
 - 9.6.2. The data acquisition system (DAS) converts the wind speed output signal (0-4.89 VDC) to wind speed in m/s.
 - 9.6.3. The DAS converts the wind direction signal (0-4.89 VDC) to wind direction in angular degrees (0-352°).
- 9.7. Computer hardware & software
- 9.7.1. The DAS provides a regulated DC excitation voltage (maximum 24 VDC) to the potentiometer.

10. Data and Records Management

- 10.1. Collect, store, and distribute data according to SOP B5 and the QAPP.
- 10.2. Store the original calibration documents in the on-site instrument shelter in the instrument folder that contains the manual and certifications.
- 10.3. Document all instrument checkouts (initial and periodic) in the electronic field notes.
- 10.4. Screen data using the criteria in Table 1.
- 10.5. Strikeout all errors in writing with a single line. Initial and date all such corrections.

11. Quality Control and Quality Assurance

- 11.1. The instrument is calibrated by the manufacturer and is shipped with a calibration certificate.
- 11.2. During the receiving inspection check, verify the wind speed and direction outputs (Section 9.2).
- 11.3. Conduct a performance check (including bearing status using the propeller torque disc) at intervals specified by the QAPP.
- 11.4. Routinely inspect data for validity, and flag data automatically according to criteria in Table 1. Investigate flagged data following procedures in SOP B5.

12. References

- 12.1. R.M. Young Co. (2001). Model 003002V Wind Sentry with Voltage Outputs Manual PN 03002V-90, Revision C060403.
- 12.2. R.M. Young Co. (2003). Wind System Calibration, Recommended Calibration Interval, Procedure, and Test Equipment. Manual PN 18860-90. PDF format.
- 12.3. US EPA, Office of Air Quality Planning and Standards. 2000. Meteorological Monitoring Guidance for Regulatory Modeling Applications. Available at <http://www.epa.gov/scram001/guidance/met/mmgrma.pdf>. Accessed on 2/10/2006.
- 12.4. SOP B1. 2006. Data Acquisition and Control Hardware. Standard Operating Procedure B1. Purdue Ag Air Quality Lab.
- 12.5. SOP B2. 2006. Data Acquisition and Control Software (AirDAC). Standard Operating Procedure B2. Purdue Ag Air Quality Lab.
- 12.6. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 12.7. SOP E5. 2006. Roof-mounted Weather Station Tower. Standard Operating Procedure E5. Purdue Ag Air Quality Lab.

13. Supplier Contact Information

R.M Young Company
 2801 Aero Park Drive, Traverse City, Michigan 49686 USA
 Tel: (231) 946-3980, Fax: (231) 946-4772
 E-mail: met.sales@youngusa.com
www.youngusa.com

Table 1. Wind speed and direction data screening criteria.

Variable	Screening criteria: Flag data if the value
Wind speed	is less than zero or greater than 25 m/s does not vary by more than 0.1 m/s for 3 consecutive hours does not vary by more than 0.5 m/s for 12 consecutive hours is zero for more than two hours consecutively
Wind Direction	is less than zero or greater than 360 degrees does not vary by more than 1 degree for more than 3 consecutive hours does not vary by more than 10 degrees for 18 consecutive hours

Reference: US EPA, 2000, Table 8.4, page 8-24.

ROOF-MOUNTED WEATHER STATION TOWER
Standard Operating Procedure (SOP) E5

ROOF-MOUNTED WEATHER STATION TOWER

Standard Operating Procedure (SOP) E5

Prepared by

Juan Carlos Ramirez

Reviewed by

Albert J. Heber, Bill W. Bogan and Kenneth D. Casey (Texas A&M Univ.)

Effective Date: November 13, 2007

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability.....	3
2. Summary of Method.....	3
3. Definitions.....	3
4. Health and Safety.....	3
5. Cautions.....	4
6. Personnel Qualifications.....	4
7. Equipment and Supplies.....	4
8. Procedure.....	5
9. Data and Records Management.....	6
10. Quality Control and Quality Assurance Section.....	6
11. References.....	6
12. Contact Information.....	7

1. Scope and Applicability

- 1.1. A commercial aluminum tapered tower is needed to support sensors for solar radiation, wind speed and direction, ambient temperature and relative humidity at a barn emission monitoring site.
- 1.2. The roof-mounted tower will be located at the ridge of the barn near the instrument shelter. The vertical distance from the ground depends on the ridge height of the barn.
- 1.3. Weather data collected using this tower will be used to help explain abnormalities registered by other instruments, such as the effect of wind direction on the performance of the barn's low-pressure exhaust fans.
- 1.4. The data will be used to model thermal characteristics of the barn including heat loss and ventilation rate.
- 1.5. The tower will be installed on barn roofs with a slope not exceeding 4:12 (rise:run).
- 1.6. This SOP applies to the Model RT-424 tapered tower (Glenn Martin Engineering, Boonville, MO) or equivalent.

2. Summary of Method

A four-legged 1.37-m (4.5-ft) high tower with a maximum width of 0.6 m (24.5 in.) will be installed on the roof of a barn, and weather instruments will be installed on the tower, as shown in Fig. 1. The tower weighs about 30 lb, and is strong enough to support weather instruments. Stainless steel hardware and fasteners are included with the tower. Independent cables will be used for the lightning ground and for grounding the sensors. The farm's employees will install the tower on the roof, and project personnel will install the sensors and the corresponding signal, power, and grounding cables.

3. Definitions

- 3.1. NEC National Electrical Code
- 3.2. OSHA Occupational Safety and Health Agency
- 3.3. PAAQL Purdue Agricultural Air Quality Laboratory

4. Health and Safety

Any work performed on the roof when installing the tower and the weather monitoring equipment should follow OSHA requirements for roof work and ladder safety, which include, as a minimum, the following aspects:

- 4.1. Inform the farm contact person to obtain authorization to work on or access the roof. Fill out any necessary forms.
- 4.2. Ask for assistance from the farm licensed electrician to install any electric cable.
- 4.3. Roof Work Safety
 - 4.3.1. Do not attempt to climb onto slippery roofs. Inspect for and remove ice and snow before getting onto roof surfaces. If the roof surface is wet, wait until the surface is dry.

- 4.3.2. Use a safety harness system with a solid anchor point on steep roofs if the ground-to-eave height exceeds 25 ft.
- 4.3.3. Do not access the roof during storms, high winds or other adverse weather conditions.
- 4.3.4. Remove or properly guard any impalement hazards.
- 4.3.5. Wear shoes with slip-resistant soles.
- 4.4. Ladder Safety:
 - 4.4.1. Keep manufactured and job-made ladders in good condition and free of defects.
 - 4.4.2. Inspect ladders before use for broken rungs or other defects so falls don't happen.
 - 4.4.3. Secure ladders near the top or at the bottom to prevent them from slipping.
 - 4.4.4. When you can't tie the ladder off, be sure the ladder is on a stable and level surface so it cannot be knocked over or have its bottom kicked out.
 - 4.4.5. Place ladders at the proper angle (4:1 slope = rise:run).
 - 4.4.6. Extend ladders at least 3 ft above the eave of the roof to provide a handhold, or for balance when getting on and off the ladder from the roof.
 - 4.4.7. Always face the ladder and maintain 3 points of contact when climbing it.
- 4.5. If you drop something while working on a roof, NEVER try to catch or stop it. Let it fall and keep your own balance secure. If you are working on the ground while someone else is on the roof, be mindful of the possibility of falling objects, and try to avoid these areas.
- 4.6. Always have someone helping you nearby.
- 4.7. Avoid installing the tower close to wires or power lines. Stay at least 1½ times the overall height away from any power lines or wires.

5. Cautions

- 5.1. Test the resistance to ground of the lightning rod before cutting the lightning rod cable (Section 8.10.2). If a second rod is necessary, the length of the grounding cable should be longer.

6. Personnel Qualifications

- 6.1. Installer and operators need to read and understand this SOP, and must be able to work in open heights without getting dizzy.

7. Equipment and Supplies

- 7.1. Tapered tower (Model RT-424, Glen Martin Engineering, Boonville, MO)
- 7.2. Mast to support the wind speed and direction sensor
- 7.3. Mast clamps (2) for non-rotating masts (Part #MC-10)
- 7.4. Side arm to install other sensors, such as the solar radiation pyranometer and the temperature and relative humidity sensors
- 7.5. U bolts to mount the side arm
- 7.6. Lightning Rod kit (Part #LR-8400) with Aluminum Tapered Point
- 7.7. Lightning cable
- 7.8. Copper grounding rod

- 7.9. Ground wire: 4 AWG solid soft bare copper wire, or equivalent
- 7.10. Battery-powered drill (to attach the bolted grounding cable to the tower)
- 7.11. Screwdriver to tighten the mast adaptor
- 7.12. Clipper to tighten the leg bolts, clamps, U bolts, etc
- 7.13. UV-resistant plastic ties to hold cables
- 7.14. Safety harness with lifeline
- 7.15. Ladder that can be extended at least three feet above the roof or landing surface

8. Procedure

- 8.1. Establish the tower location as close as possible to the instrument shelter. Make sure that trees or buildings will not cast a shadow on the solar radiation sensor that will be installed on a side arm on the tower.
- 8.2. Check that the roof presents the following conditions:
 - 8.2.1. Dry: If it is wet, dry the surface or wait until it is dry.
 - 8.2.2. Clean: Any material on the roof can create a slippery surface.
 - 8.2.3. Smooth: Warped roofs imply potential damage to the deck under the shingles.
 - 8.2.4. Secure: Verify that there are no nails or other materials that present a hazard.
- 8.3. Secure the bracket for the lifeline that will be connected to the safety harness.
 - 8.3.1. Construct roofing brackets to fit the pitch of the roof. Secure brackets in place by nailing, in addition to the pointed metal projections. Drive the nails full length into the roof (in accordance with 29CFR 1919.28(s)).
 - 8.3.2. When rope supports are used, they shall consist of first-grade manila of at least three-quarter-inch diameter, or equivalent.
 - 8.3.3. The safety belt lanyard shall be a minimum of ½-in nylon, or equivalent, with a maximum length to provide for a fall of no greater than 6 ft. The rope shall have a nominal breaking strength of 5,400 lbs.
- 8.4. Assemble the tower in accordance with the instruction manual.
- 8.5. Prepare the tower before installing it on the roof.
 - 8.5.1. Install all the instrumentation on the tower when it is on the ground, to verify that all the components are in place and to minimize the work on the roof (drilling, measurements, etc.).
 - 8.5.2. Install the U bolts so the side arm can be installed once on the roof.
 - 8.5.3. Mark which side should face south (the side arm for the solar sensor projects southward), so that the installer will clearly know the correct orientation.
 - 8.5.4. Attach the bolted fixture to which the lightning rod and cable will be installed.
 - 8.5.5. After testing sensors on the ground with the data acquisition system (as described in their respective SOPs), uninstall the sensors and have the tower ready to be lifted up by the farm employees.
- 8.6. Install the tower on the roof.
 - 8.6.1. Coordinate the installation of the tower on the roof.
 - 8.6.2. When hoisting lines are used to raise tools, materials, or instruments to a roof greater than 16 ft from ground to eaves without a parapet (or with a parapet less than 30 in. height), the installer on the roof shall be secured by an approved safety belt attached to a lifeline.

- 8.7. Install grounding rod and cable.
 - 8.7.1. Measure the distance from the tower to the location of the grounding rod.
 - 8.7.2. Check that the layout of the cable on the roof is not going to pose a trip hazard.
 - 8.7.3. Drive at least 8 ft of the rod into the earth (NEC, Sect. 250.83, 1996).
- 8.8. Install weather sensors.
 - 8.8.1. Install the wind speed and direction sensor on a non-rotating post on top of the tower. Follow SOP E4.
 - 8.8.2. Install the solar radiation sensor according to SOP E3.
 - 8.8.3. Install the temperature and relative humidity sensors, following SOP E2.
- 8.9. Install the sensors' signal cables.
 - 8.9.1. Follow each instrument's SOP for the signal and power connections.
 - 8.9.2. Tie up all these cables together.
- 8.10. Troubleshooting
 - 8.10.1. If tower becomes loose from its foundation, have the farm contact person tighten it.
 - 8.10.2. If resistance to ground of the lightning rod is higher than 25 ohms, install a second rod at a minimum distance of 6 ft, and connect the two rods (NEC, Section 250.83, 1987).

9. Data and Records Management

- 9.1. Maintain all records in the electronic field notes. Supplement this electronic record by a bound record book, which should contain copies of the electronic record.
- 9.2. Manage all data according to SOP B5.
- 9.3. Document all data and information on field data sheets located in the logbook with permanent ink, and in the electronic field notes.
- 9.4. Strike out all errors with a single line. Initial and date all such corrections.

10. Quality Control and Quality Assurance Section

- 10.1. Verify that the resistance to ground of the lightning system is below 25 ohms (in accordance with Reference 11.2, Section 250.84). If it is not, refer to Section 8.10.2.

11. References

- 11.1. NAHB-OSHA National Association of Home Builders/ U.S. Occupational Safety and Health Administration (1999). Jobsite Safety Handbook, 2nd ed. Available on line at <http://www.osha.gov/doc/jobsite/>, Accessed on January 4, 2006.
- 11.2. National Fire Protection Association. 1995. National Electric Code, 1996 Edition. National Fire Protection Association, Quincy, MA.
- 11.3. SOP E2. 2006. Measurement of Relative Humidity (RH) and Temperature with the Vaisala HMT100 Humidity Transmitter. Standard Operating Procedure E2. Purdue Ag Air Quality Lab.
- 11.4. SOP E3. 2006. The Measurement of Solar Radiation with the Licor Model 200SL Silicon Pyranometer. Standard Operating Procedure E3. Purdue Ag Air Quality Lab.

- 11.5. SOP E4. 2006. Wind Speed and Direction Measurements Using the R.M. Young Model 05103L Anemometer. Standard Operating Procedure E4. Purdue Ag Air Quality Lab.
- 11.6. SOP U2. 2006. Installation of Barn Measurements. Standard Operating Procedure U2. Purdue Ag Air Quality Lab.

12. Contact Information

- 12.1. Manufacturer: Glen Martin Engineering, Inc., 13620 Old Hwy 40 Boonville, MO 65233. (660) 882-2734. <http://www.glenmartin.com/industrial/pg17.htm>.
- 12.2. PAAQL: odor@purdue.edu, heber@purdue.edu, <http://www.AgAirQuality.com>.
- 12.2.1.



Figure 1. Weather station tower installation on a barn rooftop.

THE PAAQL GAS SAMPLING SYSTEM
Standard Operating Procedure (SOP) G1

THE PAAQL GAS SAMPLING SYSTEM
Standard Operating Procedure (SOP) G1

Prepared by

Juan Carlos Ramirez, Sam M. Hanni, and Ji-Qin Ni

Reviewed by

Bill W. Bogan and Albert J. Heber

Effective Date: November 6, 2006

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions	4
4. Health and Safety	4
5. Cautions	4
6. Interferences	4
7. Personnel Qualifications.....	5
8. Equipment and Supplies	5
9. Procedure.....	6
10. Data and Records Management	12
11. Quality Assurance and Quality Control	13
12. References.....	13

1. Scope and Applicability

- 1.1. Comprehensive measurement of gas emissions from livestock barns often requires sequential collection of air samples from multiple locations for delivery to a single gas analyzer or a set of multiple analyzers. Sharing analyzers among sampling locations reduces analytical costs and eliminates bias caused by instrument differences. A multipoint gas sampling system (GSS) facilitates sharing of the analyzers.
- 1.2. This gas sampling method is designed to sample air from inside and outside barns at confined animal feeding operations (CAFOs).
- 1.3. The GSS draws air through long sampling lines from gas sampling probes that are up to several hundred feet away and delivers the air to analyzer(s) that are less than 10 ft away in the instrument shelter.
- 1.4. This method includes auxiliary manual sampling from a selected location using bags, sorbent tubes, impingers, and canisters for off-line analysis.
- 1.5. Only 0.0005% to 0.025% of the total airflow of a barn fan is sampled by the GSS.
- 1.6. Leak checking of the GSS is facilitated with this method.
- 1.7. Delivery of calibration gases to the analyzer is facilitated with this method.

2. Summary of Method

- 2.1. The GSS in this SOP was developed by the Purdue Agricultural Air Quality Laboratory (PAAQL) in 2006 and supersedes the GSS described by Heber et al. (2006, in press). It has sampling and bypass manifolds that are arranged in a parallel configuration on a sloped mounting board (Fig. 1). Compared to the previous design, it has better gas leakage/blockage prevention, lower gas adsorption/desorption time, and more convenient operation and maintenance. A schematic diagram of the GSS, as it is integrated with the other components at a site (gas analyzers, DAC system) is presented in Fig. 2.
- 2.2. A sampling pump draws air from selected sampling locations at 4-7 L/min.
- 2.3. A bypass pump composites all unsampled probes and purges them simultaneously with a total flow of about 10 L/min.
- 2.4. The sample air is continuously delivered to a slightly positive analyzer manifold from which each gas analyzer draws continuous subsamples.
- 2.5. The sample air is optionally delivered to a manually operated valve for collecting samples for offline analysis.
- 2.6. A computer data acquisition and control system (SOP B1 and SOP B2) controls the automatic or manual sequential gas sampling through an array of independently-controlled 3-way solenoid valves in the GSS. Typically, air from only one sampling location is provided to the sampling and analyzer manifolds.
- 2.7. This GSS has built-in continuous sensors that measure pressure (positive or negative) in the sampling manifold, and the flow rate, relative humidity, and temperature of the sampling air. Sensor data is recorded by the data acquisition system (SOPs B1 and B2).
- 2.8. This GSS is enclosed and is heated by thermal energy dissipated by the pumps and solenoids. The increased GSS temperature improves adsorption/desorption of gases and helps to prevent condensation.

3. Definitions

3.1.	AI	Analog input
3.2.	CAFO	Confined animal feeding operation
3.3.	DAC	Data acquisition and control
3.4.	DAQ	Data acquisition
3.5.	DO	Digital output
3.6.	GSS	Gas sampling system
3.7.	Ma	Analyzer manifold
3.8.	Mb	Bypass manifold
3.9.	Mc	Calibration manifold
3.10.	Ms	Sampling manifold
3.11.	OFIS	On-farm instrument shelter

4. Health and Safety

- 4.1. The GSS has two 120-VAC vacuum pumps. Follow electrical safety rules when working on the pumps.
- 4.2. Air sampled from the barns may contain air pollutants with high concentrations that could cause negative health effects. Properly exhaust excess sample air to the outside of the OFIS and keep the OFIS well ventilated.

5. Cautions

- 5.1. Handle the GSS with care to avoid dropping it to the floor.
- 5.2. When transporting the GSS or storing it for a long time, seal all the openings of the solenoid valves and tubes to avoid contamination.
- 5.3. Only hand-tighten fittings inside the GSS to avoid over tightening them.
- 5.4. Each compression fitting inside the GSS has a gripper (seal) and a ferrule. Use them correctly to avoid air leakage.
- 5.5. Use a filter (Section 8.9) with about 1 mm x 1 mm mesh holes at the end of the GSS exhaust to prevent flies from entering the exhaust tubes. Make sure that the filter does not create excessive (>10 Pa) pressure drop in the exhaust.
- 5.6. Verify all power and signal connections and make sure that they are correct before using the GSS.
- 5.7. A 47-mm Teflon filter does not always stay flat when taking it out of the box. When inserting filters into the filter holders, carefully check the filter and make sure that it does not leave part of the surface area uncovered before tightening the holder. Always tighten the filter holder with the special filter holder wrenches.

6. Interferences

- 6.1. Air leaks into the system can dilute the sample gas. Maintain the GSS and perform leak checks when suspicions arise. Correct all problems discovered during leak checks.

- 6.2. Condensation of sampling air will not only introduce measurement error, but also may damage the analyzers. Stop the GSS if condensation is found in the tubes that provide sampling air to the GSS until the problem is solved.
- 6.3. Particulate matter in the sample air can cause malfunction of the solenoid valves and the mass flow meter. Verify proper installation of the in-line Teflon filter in all sampling inlets.
- 6.4. Improper conditioning of the Tedlar bags for the bag test (Section 9.10) can cause inaccurate results. Strictly follow the procedures in Section 9.10.3 to condition each bag,

7. Personnel Qualifications

- 7.1. Personnel should read and understand this SOP and be trained in the use of the GSS before initiating the procedures.
- 7.2. The operator should be trained to change air filters, to check sensors (relative humidity, temperature, and pressure), and to perform leak checks.
- 7.3. Personnel should read the Instruction of the Gas Sampling System prepared by PAAQL and delivered with the GSS and additional information and updates.

8. Equipment and Supplies

- 8.1. The PAAQL Gas Sampling System
- 8.2. Approximately 100 ft of Teflon tube (1/4" OD x 1/8" ID) for connecting the GSS to the 47-mm Teflon filters (Fig. 3)
- 8.3. Approximately 5000 ft of Teflon tube (3/8" OD x 1/4" ID) that connects the 47-mm Teflon filters at the sampling point to the 47-mm Teflon filters before the GSS (Fig. 3)
- 8.4. Approximately 15 ft of PVC tube (3/16" ID) for connecting bypass exhaust air from the GSS to outdoors
- 8.5. Approximately 15 ft of PVC tube (5/8" ID) for connecting excess sample air from the GSS to outdoors
- 8.6. Approximately 25 ft of 16-conductor 22-gage shielded or unshielded cable for connecting the solenoid control receptacle in the GSS to the DO channels in the DAC hardware. If 16-conductor cable is not available, use two pieces of 8-conductor or 10-conductor 22-gage shield or unshielded cables.
- 8.7. Approximately 25 ft of 14-conductor 22-gage shielded cable for connecting the sensor power/signal receptacle in the GSS to the AI channels in the DAC hardware and the low voltage DC power supplies. If 14-conductor cable is not available, use two pieces of 8-conductor 22-gage shielded cables.
- 8.8. Teflon filters (1- μ m pore size and 47-mm diameter) used with the in-line filter holders
- 8.9. Standard filter holders (2" dia), with discs of air conditioner filter foam ("NaturalAire" filters, or equivalent) to keep flies out of sampling lines
- 8.10. One or two 0-5 or 0-10 L/m rotameters for checking sampling air flow balance
- 8.11. Polyethylene pipe insulation with 1/2" wall thickness and different inside diameters (e.g. 1/2", 3/4") for sampling tube insulation
- 8.12. Electrical tape to seal the ends of the tubes to prevent their contamination during maintenance, testing, transportation or storage

- 8.13. Multimeter to check electrical connections
- 8.14. A pair of walkie-talkies to communicate between operators
- 8.15. Tedlar bags for the bag test. The size of the Tedlar bag depends on the sampling flow rate. Generally, a 80 L Tedlar bag will work in most all cases.

9. Procedure

9.1. Setup

- 9.1.1. Before setting up the GSS in the OFIS, open its front and back panels and carefully check for any possible damage, loose connections, tubing/wire drops, etc. Fix all observed problems.
- 9.1.2. Set up the GSS on a solid table and make its gas ports face the gas analyzers. Locate the GSS about six inches from the wall.

9.2. Sampling and exhaust tubes connection

- 9.2.1. Insert a piece of 1/4" OD Teflon tube through the tube holder on the top of the GSS (Fig. 1) to the common solenoid port that faces upward (Fig. 3). Connect the tube to the Teflon adapter in the solenoid by hand tightening the nut (Fig. 4, left). Hand-tighten the adapter (tube holder) on the top panel so that the tube does not move (Fig. 4).
- 9.2.2. Cut the 1/4" tube with a sharp tube cutter by leaving several feet above the GSS depending on the sampling tube setup inside the OFIS.
- 9.2.3. Install a Teflon filter with 1- μ m pore size in the 47-mm in-line filter holder and tighten the holder with the special filter holder wrenches.
- 9.2.4. Connect the other end of the 1/4" Teflon tube to the outlet of the Teflon filter holder. Tighten it with a wrench (this is different type of compression fitting that does not have ferrules).
- 9.2.5. Connect the inlet of the filter holder to the 3/8" OD air sampling tube (Fig. 3).
- 9.2.6. Repeat Sections 9.2.1 to 9.2.5 to connect all the sampling tubes to the GSS.
- 9.2.7. To prevent condensation in the sampling tubes, wrap the tubes between the GSS and the raceway with pipe insulation that is available in local hardware stores.
- 9.2.8. Connect a piece of 3/16" ID PVC tube to the bypass exhaust (Fig. 3), and lead the other end of the tube to outside the OFIS.
- 9.2.9. Insert a piece of 5/8" ID PVC tube through the sample exhaust hole (Fig. 3) and connect it to the barbed fitting on the top of the analyzer manifold (Fig. 5) and run the other end of the tube to outside the OFIS.
- 9.2.10. Connect the inlet tubes of the gas analyzers to the gas ports (Fig. 1), and hand-tighten the fittings.

9.3. Cable connection

- 9.3.1. There are two cable connection receptacles in the GSS. One has 16 pins and is for solenoid control. Another has 14 pins and is for sensor power and sensor signals.
 - 9.3.1.1. Connect the solenoid control cable. Refer to the GSS internal wire connection tables and drawings specified in the GSS Instructions. Refer to the DAC design and configuration described in the DAC measurement and control signal table in SOP B1. Make a table of connections (Table 1) and record it to the field note.

- 9.3.1.2. Connect a 16-conductor cable or two 8-conductor cables to the 16-pin receptacle plug based on the connection table.
- 9.3.1.3. Connect the other end of the cable(s) to the FieldPoint DO channels of the DAC hardware according to the connection table.

Table 1. Example of the record table for GSS solenoid control connection.

Solenoid #	Pin #	Cable wire color	DO channel #	Terminal #	Sampling location
S1	1	Brown	0	1	Barn 1, fan 1
S2	2	Red	1	2	Barn 1, fan 5
S3	3	Orange	2	3	Barn 1, fan 10
S4	4	Yellow	3	4	Barn 1, fan 15
S5	5	Green	4	5	Barn 1, fan 20
S6	6	Blue	5	6	Barn 1, fan 25
S7	7	Purple	6	7	Barn 2, fan 1
S8	8	Grey	7	8	Barn 2, fan 5
S9	9	White	8	9	Barn 2, fan 10
S10	10	Black	9	10	Barn 2, fan 15
S11	11	Brown/black	10	11	Barn 2, fan 20
S12	12	Red/black	11	12	Barn 2, fan 25
S13	13	Orange/black	12	13	Barn 1, inlet
S14	14	Yellow/black	13	14	Barn 2, inlet
Ground	15	Green/black			

9.3.2. Connect the sensor cable

- 9.3.2.1. Refer to the GSS internal wire connection tables and drawings specified in the GSS Instructions. Refer to the DAC design and configuration described in the DAC measurement and control signal table in SOP B1. Make a table of connections for GSS sensors (Table 2) and record it in the field notes.
- 9.3.2.2. Connect a 14-conductor cable to the 14-pin receptacle plug.
- 9.3.2.3. Connect the other end of the cable to the relevant channels of the DAC hardware.

Table 2. Example of GSS sensor connection record table.

Sensor	Pin #	Wire color	DAC channel#	Terminal #	Ground
Pressure transducer, signal	1	Brown	FP-AI-2 ch0	2	
Pressure transducer, ground	2	Red			Ground
RH Sensor, signal	3	Orange	FP-AI-2 ch1	4	
T Sensor, signal	4	Yellow	FP-AI-2 ch2	6	
RH/T sensor, ground	5	Green			Ground
Mass flow meter, signal	6	Blue	FP-AI-2 ch3	7	
Mass flow meter, ground	7	Purple			Ground
Thermocouple, blue	8	Grey	FP-TC-3 ch0	1	
Thermocouple, red	9	White	FP-TC-3 ch0	2	
Cooling fan, +24 DVC	10	Black	FP-DO-1 ch14	15	
Cooling fan, ground	11	Brown/black			Ground

9.4. Cooling fan control

- 9.4.1. The cooling fan (shown in Fig. 1) turns on when the GSS internal temperature exceeds the set point. Set the thermostat of the fan control inside the GSS to 50°C.
- 9.4.2. Follow SOP E1 to install a thermocouple inside the GSS so that its internal temperature is monitored and recorded in the AirDAC. Install the thermocouple measurement point close to the inlet of the cooling fan.

9.5. Software configuration

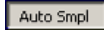
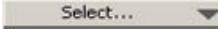

- 9.5.1. The GSS is designed to have 50% of the solenoids normally open to the sampling manifold and other 50% normally open to the bypass manifolds (Fig. 3). This is to avoid choking the pumps in case that all solenoids are off (e.g. computer shut-down or DAC power failure) but the pumps are still running.
- 9.5.2. The control of the GSS is realized by DAC hardware and software. The control configuration is set up in the DAC software, AirDAC (SOP B2).
- 9.5.3. Follow procedures specified in SOP B2 and start AirDAC. Go to page DO in AirDAC (Fig. 6) and click to enable the configuration table.
- 9.5.4. Refer to the instructions in the DO page for configuration.
 - 9.5.4.1. Enter the names of the sampling locations or other controls in the column “Name.” The Ctrl # and their matching names with the “Air smpl” control type will be automatically saved in each data file. They will also be displayed in the AirDAC for automatic or manual control as shown in the upper right corner of Fig. 6.
 - 9.5.4.2. Select control type (Ctrl type), automatic sampling sequence (Seq.), and automatic sampling frequency (Freq.). Enter the automatic sampling time (Time, m).
 - 9.5.4.3. Configure the FieldPoint digital output channels by clicking the check box table on the right side of the DO page. For instance, when controlling Ctrl # 1 (sampling location at B1, fan 1) the configuration in Fig. 6 shows that for FP-DO module 1, channels 0, and 7 through 13 will be turned on while channels 1 through 6 will be turned off.
 - 9.5.4.4. Verify that the completed configuration is correct. Click button to record the configuration to the AirDAC Config Excel file (Fig. 7). Click to disable the configuration table.
 - 9.5.4.5. If the changes need to be cancelled before applying and saving, click the button.

9.6. Power up GSS

- 9.6.1. Verify and ensure that all the completed connections are correct.
- 9.6.2. Plug the GSS power cord into a 115 VAC power outlet to power up the GSS.
- 9.6.3. Turn on the sampling pump and the bypass pump.

9.7. GSS initial test

- 9.7.1. Verify the GSS sensors
 - 9.7.1.1. Go to the DAC page in the AirDAC that is running and is also correctly configured for acquiring GSS sensor signals (Refer to SOPs B1 and B2).
 - 9.7.1.2. The pressure sensor should have values in the range between -3000 and -8000 Pa at normal sampling. If it is not in this range there may be either a leakage or a blockage in the sampling tube anywhere from the sampling location to the sampling pump.

- 9.7.1.3. The sampling flow rate should be between 4 and 7 L/m. If it is not in this range, check the sampling tubes, the sampling pump, and the mass flow meter.
- 9.7.1.4. The RH/temp sensor in the GSS should be within a reasonable range that reflects the sampling air, e.g. the RH between 20% and 70% and the T between 20°C and 45°C. Check the sensor, the sensor connection to the DAQ, and the DAQ configuration if no reasonable readings are shown.
- 9.7.1.5. The GSS temperature should show the air temperature inside the GSS.
- 9.7.2. Test solenoid control
 - 9.7.2.1. Switch to manual sampling control by clicking the  button at the upper right corner of the AirDAC. The  button appears.
 - 9.7.2.2. Click the  button and select one sampling location. When the button is released, a small clicking sound can be heard from inside the GSS, indicating that the solenoids are switching.
 - 9.7.2.3. Verify that the air sample is being taken from the selected location.
 - 9.7.2.3.1. This can be done by simply covering the inlet tube with a finger and seeing if there is suction occurring.
 - 9.7.2.4. Go through all the sampling locations and make sure that all the solenoids are configured correctly and working properly.
- 9.7.3. Verify sampling air flow balance to check for leaks in the GSS itself and in its immediate connections.
 - 9.7.3.1. This method can be used prior to leak testing of the entire sampling system (Section 9.9), since most leaks will occur in the GSS itself, or in its immediate connections. It requires two persons to work together.
 - 9.7.3.2. Check the airflow balance using two airflow (mass flow) meters, one at the inlet and another at the outlet of the sampling system.
 - 9.7.3.3. The mass flow meter inside the GSS can serve as the outlet airflow meter. Use another airflow meter or a rotameter as the inlet measurement device. Make sure that the two meters are calibrated together before the test.
 - 9.7.3.4. Connect the outlet of the airflow meter or rotameter to the inlet of the Teflon filter holder inside the barn or in the ambient air using a short piece of flexible tube.
 - 9.7.3.5. Select to sample this location in the AirDAC (Section 9.7.2) and compare the airflow readings from the air flow meter with that from the mass flow meter in the GSS. The positive difference in readings (difference = inlet - outlet) indicates a possible air leakage.
 - 9.7.3.6. If there is leakage, the leak should be identified and removed.
 - 9.7.3.7. This procedure can be combined with that of solenoid testing (Section 9.7.2) to save testing time.
- 9.7.4. Verify bypass air flow
 - 9.7.4.1. Make sure that the bypass pump has about 10 L/m flow rate and is running.
 - 9.7.4.2. Measure the airflow rate in the inlet of the filter holder at the sampling location when the line is not being sampled. Airflow rate of > 0.5 L/m should be detected, depending on the total sampling locations in the system.
 - 9.7.4.3. If the airflow rate is too small, check the flow restrictor inside the 1/4" OD Teflon tube between the bypass manifold and the solenoid connected to this

particular sampling line (Fig. 8). If it is necessary, pull out the flow restrictor from the 1/4" OD Teflon tube and cut it a little shorter (enlarge the orifice) before inserting it back into the 1/4" OD Teflon tube. Because there is a balance of bypass airflow among all sampling lines, be cautious not to enlarge the orifice of the flow restrictor too much.

9.8. GSS leakage test

9.8.1. Connect the GSS to the DAQ hardware and software so that the solenoids can be controlled, and the pressure and airflow rate can be monitored.

9.8.2. Check all air passage connections. Hand-tighten fittings inside the GSS if loose.

9.8.3. Make sure that the GSS pressure and airflow rate measurement are correct.

9.8.3.1. When the sampling pump inside the GSS is shut off, both pressure and airflow rate should read zero.

9.8.3.2. If they do not read zero, re-set them to zero by adjusting the "9. Intercept (B)" entry in tab "DAQ" in AirDAC. "Apply and save" the settings.

9.8.4. Turn the sampling location to "GSS test" that shuts off all solenoids from connecting between sampling lines and the sampling manifold.

9.8.5. Watch the readings of the sampling pressure and sampling flow rate. Both of them should drop down quickly.

9.8.6. If the sampling airflow drops to nearly 0.0 L/m within about one minute, the GSS is leakage proof starting from the solenoids. Do not leave the vacuum status too long because the GSS is "over loading" during the "GSS test."

9.9. Entire sampling system leakage test (requires two persons and a pair of walkie-talkies)

9.9.1. Turn the sampling location on one by one manually (described in Section 9.7.2) to test the leakage beyond the solenoid inlets.

9.9.1.1. Record the sampling pressure and sampling airflow rate.

9.9.1.2. Close the inlet of the filter holder at the sampling point, which is located inside or outside the barn, by a cap or simply tightly press a finger onto the inlet opening. If the sampling line does not have any leakage, the sampling airflow will drop down to close to zero within a couple of minutes, depending on the length of the tube. If the airflow is below 1% of the total sampling air flow (0.05 L/min for a 5 L/min sampling airflow), the sampling system should be considered normal.

9.9.2. Record the test results in the test notes.

9.10. Bag Test

9.10.1. Connect 3 m (10 ft) of 1/4" Teflon tubing to the output of the Environics diluter.

9.10.2. Referring to SOP G11, access the concentration mode in the Environics software, and program the diluter to produce an ammonia concentration significantly different from that which is currently being measured at the sampling location. The exact concentration of ammonia that is appropriate for this step will vary from site to site and season to season, as will the actual ammonia concentrations measured at the site. Selecting a different concentration from that which is being measured allows the operator to easily see that a particular location is being tested, and to visualize differences from one sampling location to another during the bag test.

- 9.10.3. Conditioning of the Tedlar bags
- 9.10.3.1. Check the Tedlar bags for damage that might cause leaks (holes, cuts, a bad or loose valve, etc.)
 - 9.10.3.2. Using flexible (i.e. vinyl) tubing, connect the first Tedlar bag to the vacuum pump that is used for the GSS leak test. Evacuate the bag, then disconnect it and evacuate the other 3 bags.
 - 9.10.3.3. Fill 1/3 full with ammonia gas of a known concentration for the bag test.
 - 9.10.3.4. Repeat Sections 9.10.3.2 - 9.10.3.3 two more times.
 - 9.10.3.5. Completely fill the Tedlar bag with ammonia gas of a known concentration.
 - 9.10.3.6. Repeat Sections 9.10.3.2 - 9.10.3.5 for the other 3 Tedlar bags
- 9.10.4. Test each sample location's integrity as follows:
- 9.10.4.1. Enter the AirDAC program (SOP B2).
 - 9.10.4.2. Switch to manual sampling control by clicking the button at the upper right corner of the AirDAC. The button appears.
 - 9.10.4.3. Click the button and select one sampling location.
 - 9.10.4.4. Sample that location for a minimum of 5 min before attaching the Tedlar bag with the known ammonia concentration.
 - 9.10.4.5. Using flexible (i.e. vinyl) tubing, attach Tedlar bag to the sampling location that is being measured. Use a reducing union, and make sure that the connection is a tight connection to avoid any leaks.
 - 9.10.4.6. Open valve on Tedlar bag with 3 turns. (Do not open any further than this, because the valve will come apart)
 - 9.10.4.7. Sample the Tedlar bag for 10 min.
 - 9.10.4.8. Monitor the AirDAC's gas page (Fig. 9).
 - 9.10.4.9. Save a print screen of the AirDAC's gas page in a PowerPoint file.
 - 9.10.4.10. Record the sampling time (Start & Stop), sampling location, ammonia concentration readings from the instrument & AirDAC, and the instrument model in the field notes using Table 3.

Table 3. Bag Test Table

Bag Test (30 ppm NH ₃) 11/10/2004		Bag Test TEC 17C				
Time		Sampling	Instrument	AirDAC	Average from	Percent
Start	Stop	Location	Reading	Reading	Data File	Difference
			ppm	ppm	ppm	
15:33:30	15:43:30	Fan 2	27.7	27.53	27.49	1.35%
15:54:05	16:04:00	Fan 9	27.2	27.03	26.7	4.18%
16:08:30	16:18:30	Fan 15	27.6	27.44	27.44	1.53%
16:24:08	16:34:08	Fan 22	27.2	27.14	27.12	2.67%
16:40:29	16:50:30	Fan 41	28.1	27.97	27.93	0.23%
16:56:43	17:06:56	Fan 35	29	28.84	28.82	3.43%
17:15:30	17:25:30	Fan 29	29.1	28.93	28.84	3.50%
17:43:00	17:53:00	Ambient	28.8	28.6	28.58	2.57%
		Average			27.865	2.43%

- 9.10.4.11. Switch to the next location.
- 9.10.4.12. Repeat steps 9.10.5.4 to 9.10.5.10 until all sampling locations have been tested.
If necessary, refill empty Tedlar bags with the known ammonia gas concentration again until all the locations have been tested.
- 9.10.4.13. Switch back to Auto Sampling Mode in AirDAC.
- 9.10.5. Averaging the ammonia concentrations from the 15-s data file
 - 9.10.5.1. Go to Data file
 - 9.10.5.2. Copy and paste the copy of the current 15-s data file in the data file
 - 9.10.5.3. Open the copy of current 15-s data file with Excel.
 - 9.10.5.4. Locate the start and stop times for each location during the bag test in the 15-s data file.
 - 9.10.5.5. Average the ammonia concentration readings for the last two minutes of the Tedlar bag sampling.
 - 9.10.5.6. Using Table 3, enter the average ammonia concentrations for each sampling location into the field notes.
 - 9.10.5.7. Close the copy of the 15-s data file without saving.
 - 9.10.5.8. Save the field notes.
 - 9.10.5.9. Table 3 calculates the percent difference from average ammonia concentration reading from all sampling locations for each sampling location.
 - 9.10.5.10. If the percent difference is greater than 5 % for any sampling location, take the following corrective action(s), and retest that sampling location (repeat Sections 9.10.5.4 to 9.10.5.10), until the percent difference is less than 5 %.
 - 9.10.5.10.1. Check and tighten all connections for the sampling line that has a leak.
 - 9.10.5.10.2. Check for holes or damage to the sampling line.
 - 9.10.5.10.3. Check to make sure that the filter holder closed tightly
 - 9.10.5.10.4. Check the GSS for leakage or loose connections
 - 9.10.5.10.5. Check Tedlar bag for leaks
 - 9.10.5.10.6. Check the connects between the GSS and the gas analyzers
 - 9.10.5.10.7. Check the connection between the sampling location and the Tedlar bag
 - 9.10.5.11. Check the Tedlar bags for damage that might cause leaks (holes, cuts, a bad or loose valve, etc.).
 - 9.10.5.12. Evacuate Tedlar bags and store in a safe place until needed for the next bag test.

10. Data and Records Management

- 10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
- 10.2. Manage all data according to SOP B5.
- 10.3. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes. Strike out all errors in writing with a single line. Initial and date all such corrections.

11. Quality Assurance and Quality Control

- 11.1. Review the daily measurement data for the GSS sampling pressure and flow rate to identify abnormal readings. Some common problems that can be identified by the pressure and airflow data are listed in Table 4.
 - 11.1.1. Change the filters in the filter holders inside the barns according to the QAPP.
 - 11.1.2. Conduct a leakage test using procedures described in Sections 9.9 or 9.7.3 whenever sampling tubes are relocated or connections are disturbed or changed.
 - 11.1.3. Conduct the quick GSS leak tests described in Sections 9.7.4.3 every two weeks or whenever it is considered necessary.
 - 11.1.4. Keep minimum sampling airflow about 0.5 L/m higher than the maximum sample airflow drawn by all analyzers and the manual port. If additional analyzers connected to the GSS that increase sample air demand, adjust the flow restrictor between the sampling pump and the mass flow meter to increase the sampling flow rate.

Table 4. Possible sampling system problems related to GSS pressure and airflow data.

Pressure	Airflow	Related locations	Possible problem and solution
Low	Low	One or several	An overloaded air filter or a blockage in the sampling line. Change the filter and clean the blockage.
High	Low	All locations	Sampling pump loses vacuum. Replace worn diaphragm and fix loose check valves inside pump.
High	High	One or a few	Leakage in the sampling line or GSS.
Low	High	All	Leakage in the GSS.
Unstable	Unstable	One	Solenoid not receiving stable power due to DO channel problem. Change DO channel or module.

12. References

- 12.1. Heber, A.J., J.-Q. Ni, B.L. Haymore, R.K. Duggirala, and K.M. Keener. 2001. Air quality and emission measurement methodology at swine finishing buildings. *Transactions of ASAE* 44(6):1765-1778.
- 12.2. Heber, A.J., J.Q. Ni, T.T. Lim, A.M. Schmidt, J.A. Koziel, P.C. Tao, D.B. Beasley, S.J. Hoff, R.E. Nicolai, L.D. Jacobson, and Y. Zhang. 2006. Quality assured measurements of animal building emissions: Gas concentrations. *AWMA Journal* (in press).
- 12.3. SOP A5. 2006. Measurement of Fan Static Pressure in Livestock Buildings with Setra Model 267 Differential Pressure Transducer. Standard Operating Procedure A5. Purdue Ag Air Quality Lab.
- 12.4. SOP B1. 2006. Data Acquisition and Control Hardware. Standard Operating Procedure B1. Purdue Ag Air Quality Lab.
- 12.5. SOP B2. 2006. Data Acquisition and Control Software (AirDAC). Standard Operating Procedure B2. Purdue Ag Air Quality Lab.
- 12.6. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.

- 12.7. SOP E1. 2006. Temperature Measurement Using Thermocouples. Standard Operating Procedure E1. Purdue Ag Air Quality Lab.
- 12.8. SOP U1. 2006. On-farm Instrument Shelters for Barn Sources. Standard Operating Procedure U1. Purdue Ag Air Quality Lab.
- 12.9. SOP U2. 2006. Installation of Barn Measurements. Standard Operating Procedure U2. Purdue Ag Air Quality Lab.
- 12.10. SOP U3. 2006. Heated Raceway. Standard Operating Procedure U3. Purdue Ag Air Quality Lab.

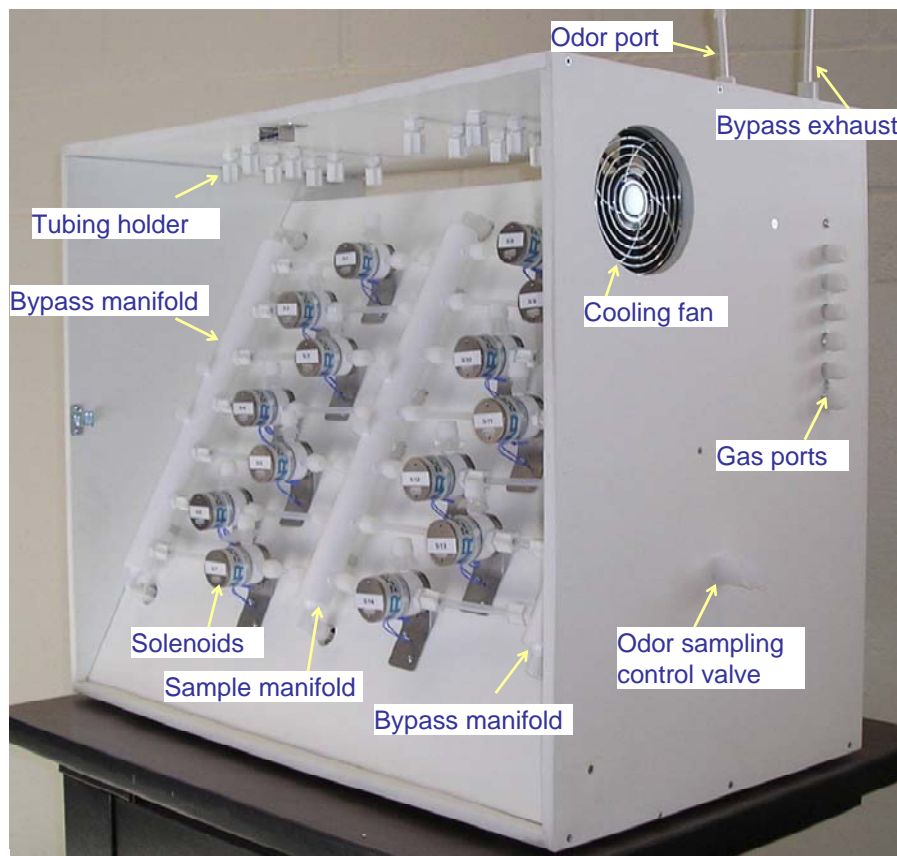


Figure 1. Open-cover view of the multi-point Gas Sampling System (front).

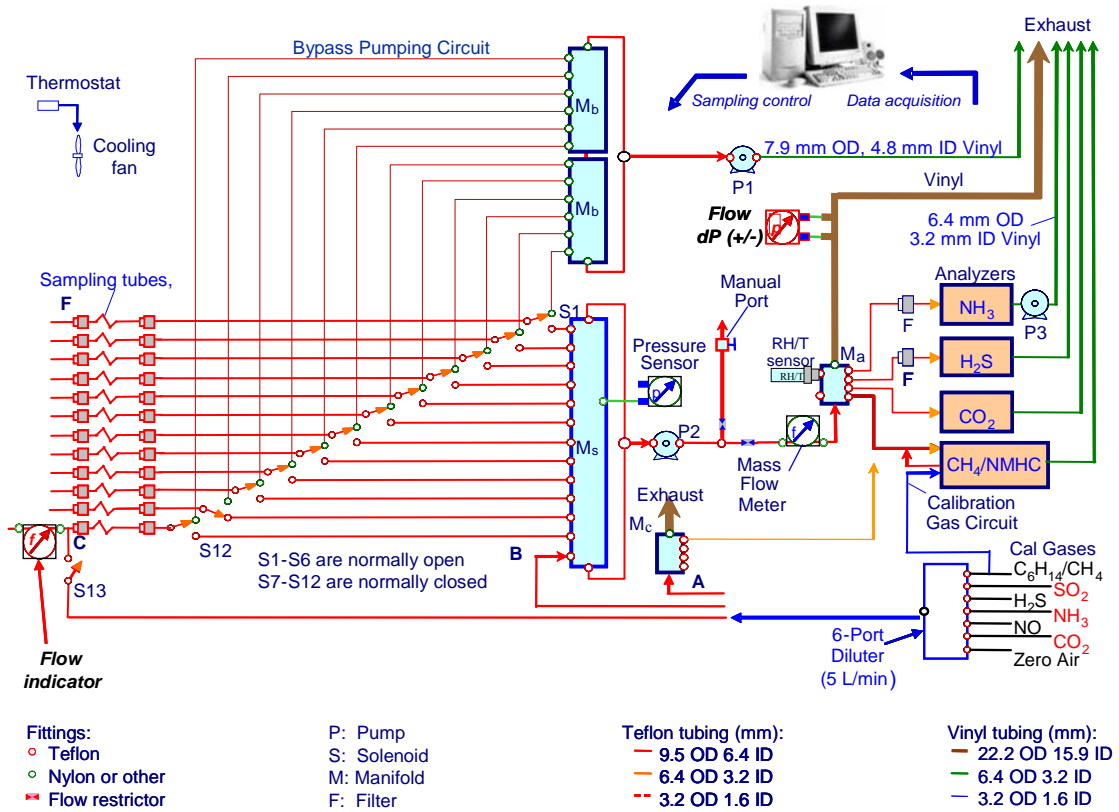


Figure 2. Schematic of PAAQL Gas Sampling System.

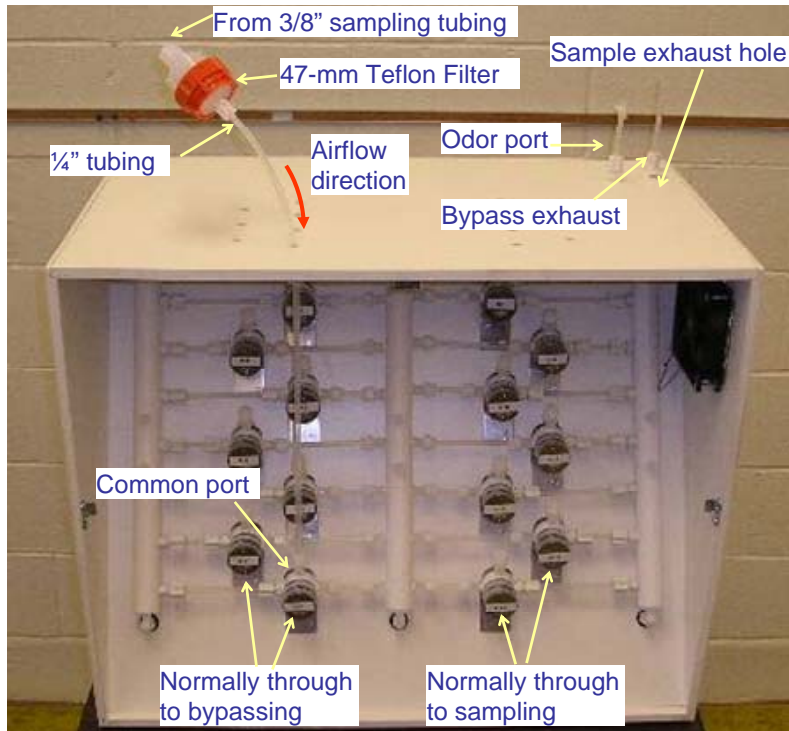


Figure 3. Connection of sampling filter holders and air inlets (only one shown as an example).

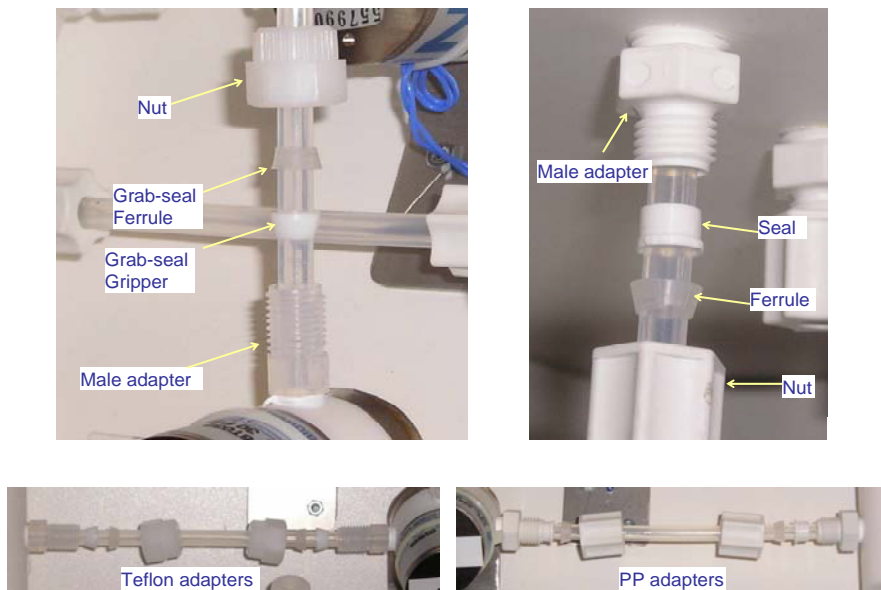


Figure 4. Connection of sampling tube to the Grab-seal Teflon fitting (left) at the solenoid and through the White PP (right) fitting as the tube holder on top panel of the GSS.

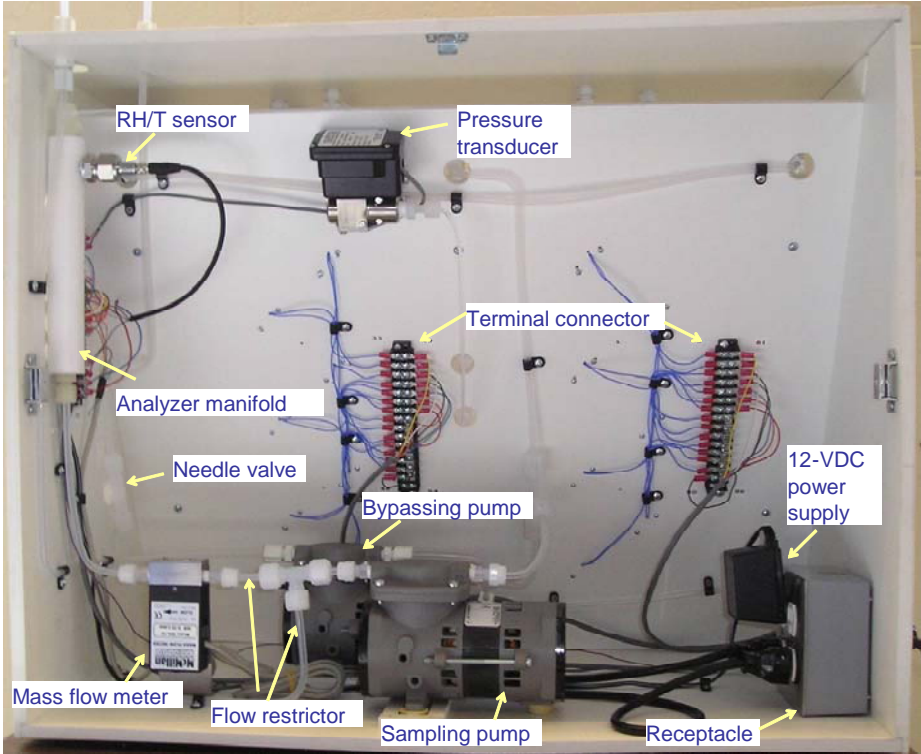


Figure 5. Open-cover view (back) of the multi-point gas sampling system.

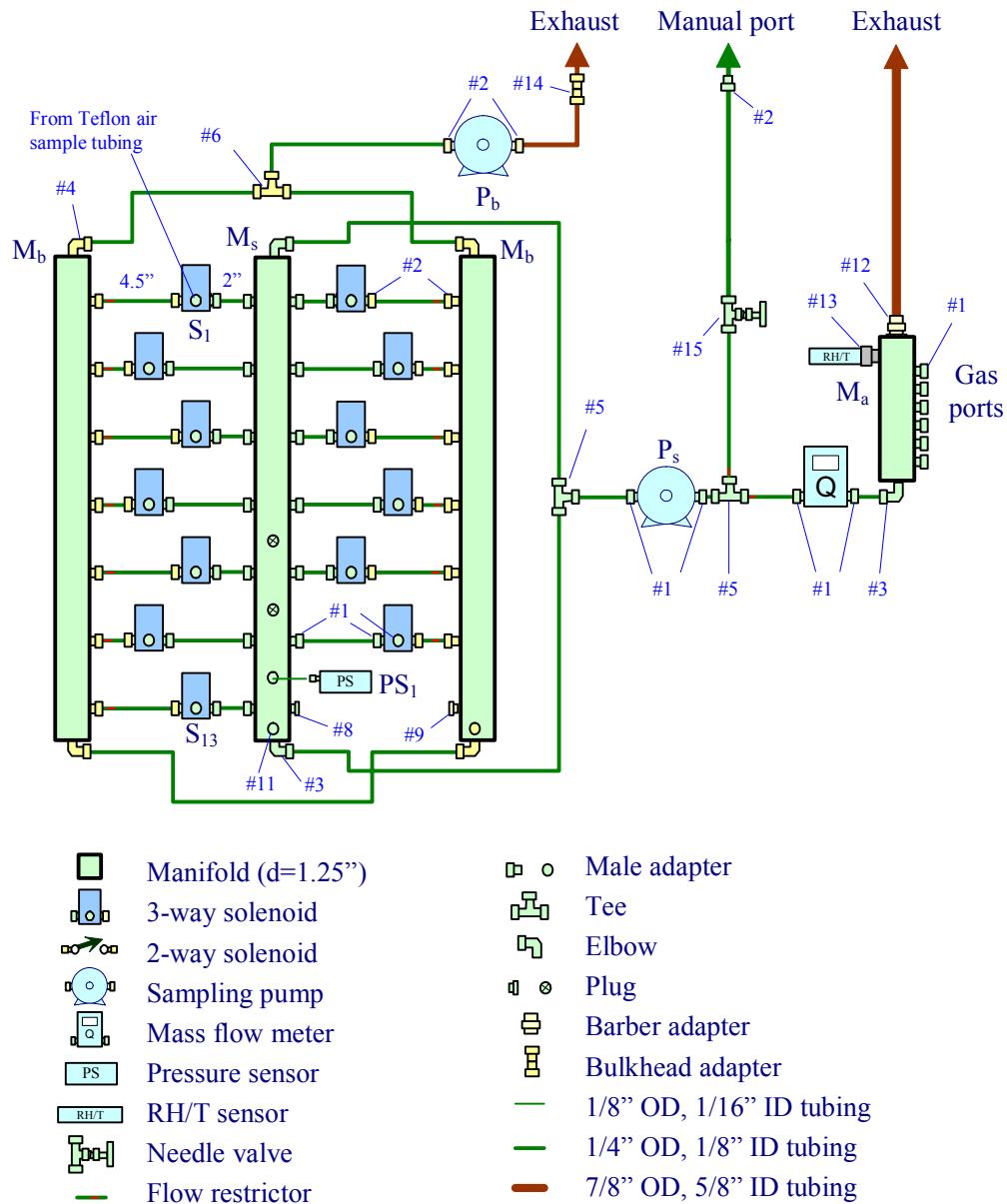
6/23/2006 10:44

Name of control	B1, fan 1	B1, fan 5	B1, fan 8	B1, fan 15	B1, fan 20	B1, fan 25	B2, fan 1	B1, inlet	B2, inlet	GSS test
Control Type	0	0	0	0	0	0	0	0	0	1
Smpl Sequence	1	2	3	4	5	6	8	7	14	0
Smpl time, min	10	10	10	10	10	10	10	30	30	0
Smpl 1 every n cycles	1	1	1	1	1	1	1	3	3	0
FP-DO 1 number	16257	16258	16260	16264	16272	16288	16320	12160	8064	16256
FP-DO 2 number	256	256	256	256	256	256	256	256	256	256

6/23/2006 10:50

Name of control	B1, fan 1	B1, fan 5	B1, fan 10	B1, fan 15	B1, fan 20	B1, fan 25	B2, fan 1	B1, inlet	B2, inlet	GSS test	B1 Raceway T
Control Type	0	0	0	0	0	0	0	0	0	1	1
Smpl Sequence	1	2	3	4	5	6	8	7	14	0	0
Smpl time, min	10	10	10	10	10	10	10	30	30	0	0
Smpl 1 every n cycles	1	1	1	1	1	1	1	3	3	0	0
FP-DO 1 number	16257	16258	16260	16264	16272	16288	16320	12160	8064	16256	16384
FP-DO 2 number	256	256	256	256	256	256	256	256	256	256	256

Figure 7. An example of the recorded AirDAC DO configuration in the Excel file. The grey-colored cells indicate latest changes.



Fitting color: Light green = Teflon; Yellow = White PP or nylon; Gray = Stainless steel
 Tubing color: Green = Teflon; Brown = other material

Figure 8. Sampling system diagram. Fitting # is specified in the GSS instructions. Note that the 14th solenoid (lower right) has been omitted to show the pressure sensor (PS) behind it.

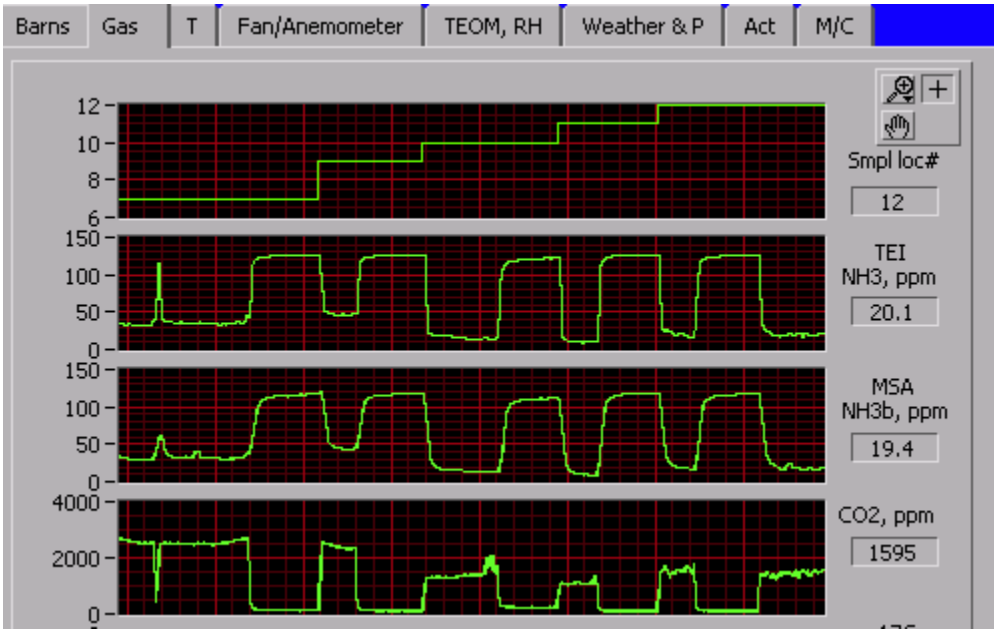


Figure 9. Responses of the two ammonia gas analyzers to a test using ammonia-filled bags at five sampling locations.

COMPRESSED GAS CYLINDERS
Standard Operating Procedure (SOP) G2

COMPRESSED GAS CYLINDERS
Standard Operating Procedure (SOP) G2

Prepared by

Congna Li, Juan Carlos Ramirez, and Claude A. Diehl

Reviewed by

Bill W. Bogan and Albert J. Heber

Effective Date: November 6, 2006

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions of Terms and Acronyms	3
4. Health and Safety	4
5. Cautions	7
6. Personnel Qualifications.....	8
7. Equipment and Supplies	8
8. Procedures	8
9. Troubleshooting	12
10. Data and Records Management	12
11. Quality Control and Quality Assurance	14
12. References.....	14

1. Scope and Applicability

- 1.1. Gas cylinders are used to transfer certified gas standards to research labs where gas concentration measurements are conducted. They are also used to supply fuel for flame ionization detectors, carrier gases for gas chromatographs, dilution gases for programmable diluters, and concentrated gases for gas injection systems. The following general facts about compressed gas cylinders are relevant to this SOP:
 - 1.1.1. Gas standards typically have a defined, finite shelf life, and a specific expiration date.
 - 1.1.2. Gas cylinders contain a finite volume of gas that may need to be replenished.
 - 1.1.3. Gas cylinders are typically rented so money is saved by returning them promptly.
 - 1.1.4. Gas cylinders generally look alike which challenges identification procedures.
 - 1.1.5. Missing cylinders must be replaced at a significant replacement charge.
 - 1.1.6. The time between purchase and delivery are relatively long.
 - 1.1.7. The high pressure inside a gas cylinder represents a potential safety hazard.
 - 1.1.8. The contents of a gas cylinder can range from very safe to extremely hazardous.
 - 1.1.9. A gas-specific pressure regulator is required for each cylinder.
 - 1.1.10. Managing gas cylinders requires a significant amount of labor.
 - 1.1.11. They may be exposed to the extremes of indoor and outdoor temperatures.

2. Summary of Method

This SOP covers the necessary procedures, cautions and documentation for selecting, purchasing, receiving, transporting, storing, using, terminating, and returning gas cylinders. The compressed gas cylinder inventory will be scientifically selected, carefully controlled, and professionally handled to reduce cost, ensure safety, and improve data quality. Gas regulators will be properly selected and installed on each cylinder to prevent leakage and ensure safe delivery of the gases. Following this method will ensure proper documentation of the cylinders and the regulators that are used with the cylinders.

3. Definitions of Terms and Acronyms

- 3.1. ABE: Agricultural and Biological Engineering
- 3.2. CGA: Compressed Gas Association.
- 3.3. PAAQL: Purdue Agricultural Air Quality Laboratory
- 3.4. REM: Office of Radiation and Environmental Management at Purdue University
- 3.5. Compressed Gas: Any material or mixture having in the container either an absolute pressure exceeding 40 psia (3 bar) at 70°F (21°C).
- 3.6. Requested Concentration: The gas concentration requested by the customer when purchasing a standard gas.
- 3.7. Balance Gas: The material ("component") making up the majority of a mixture.
- 3.8. Blended Concentration: The concentration measured by the blending process. The numerical concentration assigned to a component based on the results of blending.

- 3.9. Blend Tolerance: The degree of agreement between the Blended Concentration and the Requested Concentration.
- 3.10. Crawl: Rise above a set delivery pressure during operation of a regulator.
- 3.11. Accuracy: The statistical agreement of a measured value with its true value
- 3.12. Expiration date: The stated date after which a gas concentration is no longer valid or legal to use for calibration purposes.
- 3.13. Traceability: Basis for establishing the true value of a standard or pathway back to the true value. For example, it is the relationship between a measured value and an established element of the National/International Measurement System.
- 3.14. Two-Stage Pressure Reducing Regulator: A regulator which reduces high-pressure to low pressure and controls the low or outlet pressure with two stages of pressure reduction. Used when more stability of operation is required.
- 3.15. Stability: The ability to maintain a constant concentration value over a defined time within statistical significance
- 3.16. Gas Mixture Grades
 - 3.16.1. EPA Protocol Grade: EPA Protocol Gases are manufactured and analytically certified in strict accordance with the most recent EPA traceability guideline document entitled “EPA Traceability Protocol for Assay and Certification of Gaseous Standards”. EPA protocol certified standards meet or exceed gas standards specified in U.S. regulations 40CFR60 and 40CFR75.
 - 3.16.2. Primary Grade: Specialty gas, high resolution, gravimetric mixture with certification accuracy of either 1% relative, or 0.02% absolute, whichever is less. Often referred to as NIST-Traceable By Weight.
 - 3.16.3. Certified Grade: Working standards prepared either by partial pressure or gravimetrically. Certification accuracies may range from $\pm 2\%$ to $\pm 5\%$.
 - 3.16.4. Unanalyzed Grade: The unanalyzed grade is used when the minor component accuracy does not have to be less than 10%.

4. Health and Safety

- 4.1. Always use the smallest size cylinder required to perform the work.
- 4.2. Cylinders of compressed gases must be handled as high potential energy sources.
- 4.3. Cylinders on wheeled carts must be capped and secured by an approved cylinder support strap or chain. The cart must be an approved cylinder cart. Do not attempt to take a loaded cylinder cart up or down a stairway.
- 4.4. Secure uncapped cylinders independently to a solid element of the lab structure. Do not “gang” cylinders behind a single chain, and do not use carts to support uncapped or in-use cylinders.
- 4.5. Transporting cylinders
 - 4.5.1. Move cylinders by hand trucks or carts that are designed for this purpose. Always use a cylinder cart equipped with a chain restraint.
 - 4.5.2. During transportation, properly secure cylinders to prevent them from falling or striking each other.
 - 4.5.3. Do not move a cylinder with a regulator connected to it.
 - 4.5.4. Never transport a gas cylinder without its valve protection cap firmly in place.

- 4.5.5. Keep both hands on the cylinder cart during transport.
- 4.5.6. Follow applicable federal and state regulations when transporting cylinders on open roads. Provided that no more than 440 lbs (>220lbs of flammable gas and >220lbs of other gases including container weights) are being transported, the gases are classified as “Materials of Trade”, and are not subject to placards and other restrictions that would normally be associated with transporting larger quantities of compressed gas.
- 4.6. Securing cylinders
 - 4.6.1. Place cylinder in an upright position against a cylinder rack located against the wall.
 - 4.6.2. Place the straps around the cylinder.
 - 4.6.3. Insert the strap through the buckle and pull tight.

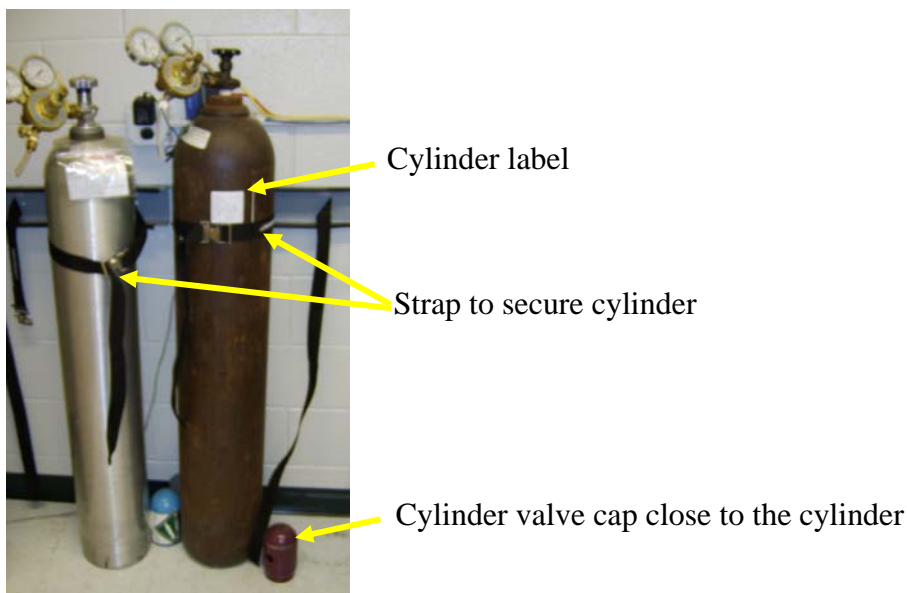


Figure 1. Gas cylinders safely located for use.

- 4.7. Storage of cylinders
 - 4.7.1. Storage Area. Store gas cylinders in a ventilated and well-lit area, away from combustible materials. Store cylinders containing flammable gases away from oxygen cylinders and other oxidants. Separate by a fire-resistant wall, or locate them at least 20 feet apart from each other.
 - 4.7.2. Storage Area Conditions. Storage areas should be located away from sources of excess heat, open flame or ignition, and not located in closed or subsurface areas. The area should be dry, cool and well-ventilated.
 - 4.7.3. Securing Cylinders in Storage. Always hold cylinders in place with a chain or another type of fastener such as a bench or wall clamp. While in storage, cylinder valve protection caps must be firmly in place.
 - 4.7.4. Cylinders may be stored flat on the ground, if vertical storage is not possible, provided they are secured as described in 4.7.3.
 - 4.7.4.1. Note that this applies only to storage. Cylinders may not be transported or used while horizontal.

- 4.7.5. For cylinders at remote sites, the conditions in 4.7.1 and 4.7.2 will be followed as closely as possible. Sections 4.7.3 and 4.7.4 will be followed strictly, including at remote sites.
- 4.8. Never tamper with safety devices in valves or cylinders.
- 4.9. Use only a cylinder cap wrench to loosen and unscrew cylinder caps; do not strike cylinder caps when attempting to loosen them.
- 4.10. Oil or grease on the high pressure side of an oxygen cylinder can cause an explosion. Do not lubricate an oxygen regulator or use a fuel gas regulator on an oxygen cylinder.
- 4.11. Wear goggles or safety glasses with side shields when handling compressed gases.
- 4.12. Eyewash fountains, safety showers, gas masks, respirators, and/or resuscitators should be located nearby but out of the immediate area that is likely to become contaminated in the event of a large release of gas.
- 4.13. Fire extinguishers, preferably a combination of water- and CO₂-based types, should be kept close at hand, and should be checked periodically to ensure their proper operation.
- 4.14. Always use appropriate gauges, fittings, and materials compatible with the particular gas. Regulators must be compatible with gas cylinders (do not use adapters). Each cylinder and regulator have connection fittings that are designated by a CGA number. CGA numbers can be stamped on the cylinder and regulator and listed on the producer's label. CGA numbers are typically (but not always) stamped on the regulator just above the threads of the cylinder connection (Fig. 2). Table 1 shows some examples of CGA connection numbers and some examples of the gases used with these connections.
- 4.15. MSDS information sheets will be kept in a 3-ring binder located near the cylinders.
- 4.15.1. MSDS information will also be kept on-site for any gases which are stored and/or used at remote locations.

Table 1. Cylinder Valve Outlets and CGA Connections.

Examples of Gases	CGA Connection #
Ammonia	705
Carbon Dioxide	320
Butane, ethylene oxide, propane, propylene	510
Air (industrial grade)	590
Carbon monoxide, ethylene, methane, hydrogen	350
Hydrogen sulfide, hydrogen chloride	330
Nitrogen, helium, argon, noble gases	580
Nitrogen dioxide, sulfur dioxide	660
Oxygen	540

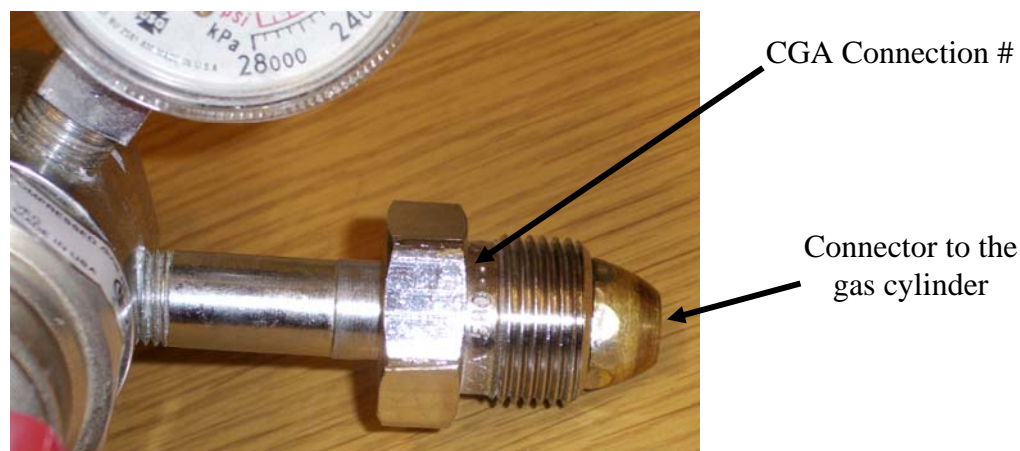


Figure 2. Photo of a gas pressure regulator (CGA 580) showing the connector to the gas cylinder. The CGA number can be found on the side of the nut closer to the regulator.

- 4.16. When work is planned with toxic, corrosive, or reactive gases, consult the MSDS and/or institutional safety office for information concerning specific handling requirements for the gas involved. Generally, these gases will need to be used and stored with local exhaust ventilation, such as a lab hood, gas cabinet, or a ventilated OFIS that is designed for that purpose.
- 4.17. Check the regulator and cylinder valve for evidence of damage or contamination.
- 4.18. Do not apply Teflon tape between the regulator and the cylinder connections. Bits of Teflon tape can get blown into the regulator, causing a leak, valve malfunction or erroneous reading.
 - 4.18.1. If a leak-free connection cannot be established between the regulator and the cylinder, inspect the regulator and cylinders threads for possible damage or contamination. If regulator components (adapter threads, washers, and/or fittings) are damaged, they should be replaced. Any regulator which cannot be made leak-free should be replaced.
- 4.19. If an OFIS at a field site (farm) contains cylinders, notify the producer and/or appropriate staff that there are compressed cylinders within the OFIS and describe the contents of the compressed cylinders. This is especially important in case of a fire, so that the producer will stay away from the OFIS, and properly notify responding emergency personnel.

5. Cautions

- 5.1. Do not allow cylinder temperature to exceed 125°F (52°C), or to drop to -30°C or lower, since many types of steel will lose their ductility and impact strength at lower temperatures.
- 5.2. Store empty cylinders separately from full cylinders, and clearly identify them as such. Leave some pressure (>50 psi) in a depleted cylinder to prevent air backflow that would allow moisture and contaminants to enter the cylinder.

- 5.3. Flammable gases have reverse threads, meaning that the connection is tightened by turning the nut counterclockwise. You can always tell a reverse thread connection because the nut that you tighten has a line inscribed around its circumference.
- 5.4. Do not use an expired cylinder for a Level 1 Precision Check. In other words, never adjust an analyzer based on data obtained from an expired cylinder.

6. Personnel Qualifications

- 6.1. Personnel should be trained in the handling of cylinders and also in the updating of the web-based cylinder control file before initiating the procedure alone.
- 6.2. Personnel handling any compressed gas should be familiar with the potential hazards before using the gas.
- 6.3. Personnel should address potential emergencies and the safe and proper measures necessary to avoid these emergencies.
- 6.4. Personnel should identify several scenarios that could result in gas leaks or other emergencies to be totally prepared to respond adequately.

7. Equipment and Supplies

- 7.1. Gas cylinders
 - 7.1.1. EPA maintains a web site which documents the performance of major gas-cylinder vendors, based on the results of a round robin test conducted every two years.
 - 7.1.2. Consider the following parameters when selecting cylinders:
 - 7.1.2.1. Certification accuracy
 - 7.1.2.2. Blend tolerance
 - 7.1.2.3. Shelf life
 - 7.1.2.4. Delivery time
 - 7.1.2.5. Reanalysis tolerance
 - 7.1.2.6. Requested concentration
- 7.2. Gas regulators appropriate for each gas (Section 4.14)
- 7.3. Labels to identify each cylinder (Section 8.1.7)
- 7.4. An adjustable or combination cylinder wrench to connect and disconnect the regulator to the cylinder.
- 7.5. Cylinder hand truck
- 7.6. Cylinder brackets
- 7.7. Cylinder return labels (Section 8.8.4)

8. Procedures

- 8.1. Receiving gas cylinders (PAAQL)
 - 8.1.1. Cylinders are delivered to the ABE shop's gas cylinder receiving station by Purdue's Gas Cylinder Control Office.
 - 8.1.2. Upon delivery, the cylinders should be picked up by PAAQL staff, after verifying that there is a plastic seal on the cylinder valve. If there is no seal, make a note and contact the company.

- 8.1.3. Write the cylinder-stamped number on the form by the gas cylinder rack, and sign the form to indicate that the cylinder has been received.
- 8.1.4. A delivery sheet or packing slip is attached to every cylinder to verify that the proper cylinder is delivered (gas type, amount of gas, cylinder ID).
- 8.1.5. Sign and date the delivery sheet.
- 8.1.6. Place the original delivery sheet in the ABE Business Office's inbox and a copy of the delivery sheet in the loose leaf cylinder notebook in the section corresponding to the year received and the company that supplied the cylinder. This notebook is kept on top of the manual's file cabinet in room ABE-102E.
- 8.1.7. Attach a cylinder label (Fig. 3) to the cylinder upon receipt, with the following information filled out:

Gas _____
Concentration _____
Initial Pressure _____ psi
CGA No. _____
Cylinder No. _____
Date Received _____
Expiration Date _____
Date: _____ Pressure: _____ psi
Date: _____ Pressure: _____ psi
Date: _____ Pressure: _____ psi
Date: _____ Pressure: _____ psi
Date: _____ Pressure: _____ psi

Figure 3: A cylinder label.

- 8.2. Receiving gas cylinders (Field sites)
 - 8.2.1. Cylinders are delivered to a specified location (e.g. the field site itself, a subcontracting university or other entity, etc) by the supplier's local vendor/agent.
 - 8.2.2. Upon delivery, the cylinders should be picked up by study personnel (PAAQL or otherwise), after verifying that there is a plastic seal on the cylinder valve. If there is no seal, make a note and contact the company.
 - 8.2.3. If necessary, transport the cylinder to the field site, observing all cautions listed in Section 4.5.
 - 8.2.4. Write the cylinder-stamped number on the form by the gas cylinder rack and sign the form to indicate that the cylinder has been received.
 - 8.2.5. A delivery sheet or packing slip will be attached to every cylinder by the supplier to verify that the proper cylinder is delivered (gas type, amount of gas, cylinder ID).
 - 8.2.6. Sign and date the delivery sheet.
 - 8.2.7. File a copy of the delivery sheet on-site in a binder or loose-leaf notebook (Section 8.1.6).

- 8.2.8. Fill out a cylinder label (Fig. 3), and attach it to the cylinder.
- 8.3. Installing pressure regulators
- 8.3.1. Make sure the cylinder is properly secured, and that the regulator is the correct one for the cylinder, and that any special hazards of the gas are known.
- 8.3.2. Remove the cylinder valve cap (counterclockwise). Place it somewhere nearby.
- 8.3.3. Some regulators (on lecture bottles and certain corrosive gases) require a Teflon washer to be inserted between the tank outlet and regulator. Check to see if this is required before continuing to the next step.
- 8.3.4. Make sure that the regulator's needle valve (A) (Fig. 4) is shut. Screw it clockwise until it seats. Do not over tighten it or you can damage the valve seat.
- 8.3.5. Make sure that the regulator's pressure adjusting handle (B) (Fig. 4) is shut. Turn it counterclockwise until it is almost completely unscrewed. If you unscrew it completely, just put it back in.

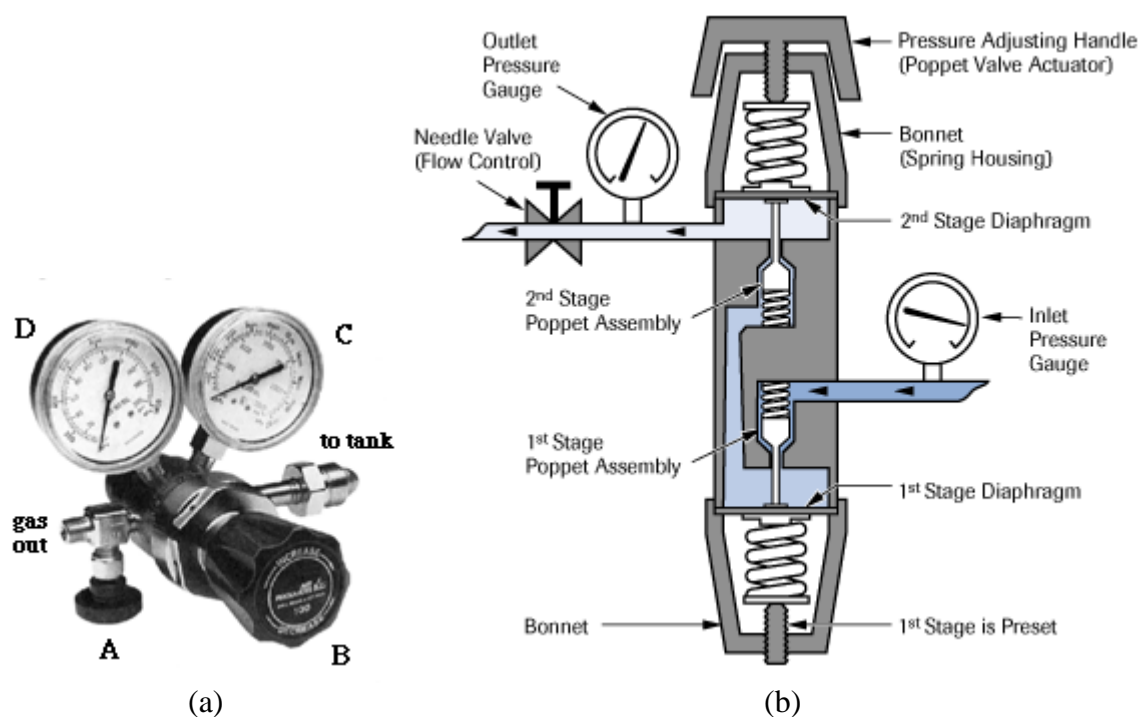


Figure 4. (a) A two –stage pressure regulator and its components. A: Needle valve for outlet flow control. B: Pressure adjusting handle (Poppet Valve Actuator). C: Inlet pressure gauge. D: Outlet pressure gauge. (b) Schematic diagram of a two-stage pressure regulator.

- 8.3.6. Turn the regulator onto the tank by hand until it is almost finger tight.
- 8.3.7. Use an adjustable wrench or cylinder wrench to tighten the regulator onto the cylinder.
- 8.4. Operation of pressure regulator
- 8.4.1. Open the tank valve slowly (counterclockwise (CCW)). Watch the tank pressure on the regulator's inlet pressure gauge (C) (Fig. 4).

- 8.4.2. Slowly turn the pressure adjusting handle (B) on the regulator until the regulator's outlet pressure gauge (D) is at the desired pressure level (Fig. 4).
- 8.4.3. Open the regulator's needle valve (A) to regulate flow with this valve.
- 8.4.4. Check your system for leaks using Snoop (a commercial product) or some soapy water. Snoop is preferred since it leaves no residue.
- 8.5. FTIR validation of gas concentration
 - 8.5.1. If the composition of the gas in a cylinder is to be checked by FTIR, this procedure should be conducted according to SOP G12 ("FTIR Verification of Gas Cylinder Composition").
- 8.6. Shut down of pressure regulator
 - 8.6.1. Shut the tank valve on the gas cylinder.
 - 8.6.2. Slowly open the needle valve (A) on the regulator.
 - 8.6.3. Watch the pressure gauges C and D drop to zero.
 - 8.6.4. Open the regulator's pressure adjusting handle (B) (turn it clockwise) to ensure that all pressure is released.
 - 8.6.5. After relieving all the gas pressure, turn the regulator pressure adjusting handle (B) CCW as far as it will go to close the regulator, and also close the needle valve (A).
- 8.7. Disconnecting a pressure regulator
 - 8.7.1. Use a wrench to disconnect the regulator from the gas cylinder.
 - 8.7.2. Replace the protective cylinder cap immediately.
- 8.8. Cylinder return
 - 8.8.1. Prior to returning the cylinder, place a return label on the cylinder and date it. (Fig. 5)
 - 8.8.2. The cylinder should be returned to the cylinder collection rack in the ABE shop.
 - 8.8.3. Place an "Empty Cylinder" ring on the cylinder.
 - 8.8.4. Enter the returned cylinder number and date on the form.

<p>Return To: General Stores</p> <p>From: Al Heber Department of Ag and Bio Engineering 1146 ABE</p> <p>Date:</p>
--

Figure 5: Return label for gas cylinders.

9. Troubleshooting

- 9.1. Check regulators periodically to ensure proper and safe operation. This periodic check will vary depending on gas service and usage. The procedure for checking out any regulator is as follows:
 - 9.1.1. Be sure gauges read zero when all pressure is drained from the system.
 - 9.1.2. Open the cylinder valve and turn the pressure adjusting handle counterclockwise; the inlet pressure gauge will read the cylinder pressure.
 - 9.1.3. Starting at Section 9.1.1, close the cylinder valve after reading the cylinder pressure, if the pressure drops after one (1) hour of monitoring the inlet pressure, there is a leak between the inlet pressure gauge and the cylinder valve.
 - 9.1.4. Starting at Section 9.1.1, close the regulator's outlet needle valve then wait 1 hour; the outlet pressure gauge should not indicate a pressure increase. A pressure increase would indicate leakage across the internal valve system.
 - 9.1.5. Turn the pressure adjusting handle clockwise until a nominal delivery pressure is indicated. Inability to attain a proper delivery-pressure setting or abnormal adjustment of the screw indicates improper operation, which may be attributed to blockage of the gas passage or a leak in the low-pressure side of the regulator. Continued wear on a regulator valve-and-seat assembly will cause a rise above a set delivery pressure, termed *crawl*. A regulator exhibiting crawl should not be used.
 - 9.1.6. Close cylinder valve and observe pressure on both inlet and delivery sides of the regulator after 1 h. A drop in the pressure reading after this period of time may indicate a leak in the system, possibly at the inlet or through the needle valve, safety devices, or diaphragm.
 - 9.1.7. An excessive fall in delivery pressure under operating conditions and normal flow indicates an internal blockage.
 - 9.1.8. Any deviation from the normal in the preceding checkout will require servicing by reputable repair personnel.

10. Data and Records Management

- 10.1. Use the information on the delivery sheet to update the PAAQL Cylinder Inventory webpage. To update this webpage you must:
 - 10.1.1. Open the "odor" drive (permission to access this drive is required).
 - 10.1.2. Open the "public-web" folder.
 - 10.1.3. Open the "cylinder" folder
 - 10.1.4. Open the "PAAQL Cylinder Inventory.htm" file.
 - 10.1.5. To edit the "PAAQL Cylinder Inventory.htm" webpage:
 - 10.1.5.1. Click on "File".
 - 10.1.5.2. Click "Edit with Microsoft Excel".
 - 10.1.5.3. Add the following data to the PAAQL Cylinder Inventory web page:
 - 10.1.5.3.1. Gas name
 - 10.1.5.3.2. Balance gas
 - 10.1.5.3.3. Gas analyzer that cylinder is assigned for calibration
 - 10.1.5.3.4. Cylinder number (this is the engraved number only)

- 10.1.5.3.5. Cylinder style
- 10.1.5.3.6. Cylinder volume
- 10.1.5.3.7. Supplier of the gas cylinder
- 10.1.5.3.8. Purchase order number
- 10.1.5.3.9. Person who requested the order
- 10.1.5.3.10. Date issued
- 10.1.5.3.11. Date received
- 10.1.5.3.12. Date tested
- 10.1.5.3.13. Date returned
- 10.1.5.3.14. Cylinder expiration date
- 10.1.5.3.15. Initial pressure, psi
- 10.1.5.3.16. Final pressure, psi
- 10.1.5.3.17. CGA number (this can be found on the cylinder valve)
- 10.1.5.3.18. Gas concentration,
- 10.1.5.3.19. Certified accuracy, %
- 10.1.5.3.20. Project
- 10.1.5.3.21. Location
- 10.1.5.3.22. Cylinder cost
- 10.1.5.3.23. Monthly cost
- 10.1.5.3.24. Total cost to date
- 10.1.5.4. Update the following data each month on the PAAQL Cylinder Inventory:
 - 10.1.5.4.1. Location
 - 10.1.5.4.2. Pressure test
 - 10.1.5.4.3. Pressure test date
 - 10.1.5.4.4. Monthly cost (see demurrage report)
 - 10.1.5.4.5. Total cost to date (see demurrage report)
- 10.1.5.5. The PAAQL Cylinder Inventory web page is color coded.
 - 10.1.5.5.1. The cylinders that have been recently returned have a purple color.
 - 10.1.5.5.2. Cylinders that are in ABE are in yellow.
 - 10.1.5.5.3. Cylinders that are on site are in dark blue.
 - 10.1.5.5.4. Cylinders not currently in the inventory but still on the demurrage report are in red.
 - 10.1.5.5.5. Cylinders that were apparently returned but yet cylinder rental charges are accruing are in green.
- 10.2. Initiate an order for a replacement cylinder well in advance of a cylinder's running out (i.e. dropping below 150 psi, as per Section 5.4).
 - 10.2.1. Each time a cylinder is used, record the pressure remaining at the end of use. Keep a graph tracking pressure vs. date to allow projection of when pressure will reach the 150 psi minimum limit.
 - 10.2.2. Allow sufficient time to meet the following requirements:
 - 10.2.2.1. Replacement cylinders will be compared to the old cylinder using the applicable gas analyzer. Leave sufficient gas in the old cylinder to conduct this test.
 - 10.2.2.2. If the replacement cylinder is outside the acceptable specifications, there must be sufficient gas remaining in the old cylinder to operate until an acceptable cylinder is received.

10.2.2.3. In general, a new purchase order should be initiated two months in advance before the current cylinder is empty or expires, if such gas is needed on a continuous basis.

10.2.3. Email notification of new order will be sent to the personnel responsible for the project.

11. Quality Control and Quality Assurance

- 11.1. Personnel handling any compressed gas will be trained to be aware of potential hazards before using the gas, to address potential emergencies and the safe and proper measures necessary to avoid these emergencies, and to identify several scenarios that could result in gas leaks or other emergencies to be totally prepared to respond adequately.
- 11.2. Regulators and cylinder valves should be checked periodically to ensure proper and safe operation.
- 11.3. The PAAQL Cylinder Inventory webpage will be used to update the information for all the cylinders in the current inventory and the most recent cylinders that have been returned.
- 11.4. Do not calibrate with a span gas if its cylinder pressure is below 150 psi.

12. References

- 12.1. Purdue University, "Chemical Hygiene Plan and Hazardous Materials Safety Manual, Version 2003.06.a revised June 2003. Retrieved from <http://www.purdue.edu/rem/home/booklets/CHP2003.pdf>
- 12.2. Shugar G.J. and Ballinger J.T. (1996). *Chemical Technician's Ready Reference Handbook*, 4th edition, McGraw-Hill, Inc. p65-79.
- 12.3. *Design and Safety Handbook for Specialty Gas Delivery Systems*. Scott Specialty Gases, 2004.
- 12.4. *Gas and Equipment Catalogue*, Matheson Tri Gas Company, 2004.
- 12.5. Interactive Learning Paradigms. The Glassware Gallery: Gas Regulators. <http://www.ilpi.com/inorganic/glassware/regulators.html>. Accessed online 12/27/2005.

**MEASUREMENT OF CARBON DIOXIDE (CO₂) WITH THE
MSA MODEL 3600 INFRARED GAS MONITOR**

Standard Operating Procedure (SOP) G3

**MEASUREMENT OF CARBON DIOXIDE (CO₂) WITH THE
MSA MODEL 3600 INFRARED GAS MONITOR
Standard Operating Procedure (SOP) G3**

**Prepared by
Ji-Qin Ni and Juan Carlos Ramirez**

**Reviewed by
Bill W. Bogan, Frank M. Mitloehner (Univ. of California-Davis), and Albert J. Heber**

Effective Date: November 6, 2006

**PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)
Agricultural and Biological Engineering, Purdue University
West Lafayette, IN 47907**

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions of Terms and Acronyms	3
4. Health and Safety	4
5. Interferences	4
6. Personnel Qualifications.....	4
7. Equipment and Supplies	4
8. Procedure.....	5
9. Data and Records Management	7
10. Quality Control and Quality Assurance	7
11. References.....	7
12. Contact Information	8

1. Scope and Applicability

- 1.1. Measurements of carbon dioxide (CO₂) in confined animal buildings (CAB) is a useful means to determine if the buildings are properly ventilated. Excess (i.e. above atmospheric) CO₂ indicates that the building is not being ventilated well enough to remove metabolically-generated gases.
- 1.2. The photoacoustic method is useful for measuring CO₂ in CAB because of the low interference of water vapor compared with NDIR.
- 1.3. Detection limit is 50 ppm. This instrument is not degraded in the presence of silicon or sulfur compounds, the last of which are often present at many CAB.
- 1.4. The range at livestock facilities is from normal atmospheric concentrations (typically 375-380 ppm) to 10,000 ppm inside the barns.
- 1.5. This procedure applies only to the MSA Model 3600 Infrared Gas Monitor.
- 1.6. The short-term stability is $\pm 1\%$ and the long term including linearity is $\pm 5\%$ in 18 months. Its response time is 12 s to read 97% of a step change in CO₂ concentration.

2. Summary of Method

Carbon dioxide (CO₂) concentration in air is measured using photo-acoustic infrared detection with a commercial analyzer (Model 3600 Infrared Gas Monitor, Mine Safety Appliances, Pittsburgh, PA). With this method, a gas sample is drawn continuously at a constant flow rate of 0.8 L/min taken from the GSS analyzer manifold (SOP G2), and introduced into an acoustic cell, where it is exposed to pulsing infrared light of specific wavelength. If the sample contains the gas of interest, it will absorb an amount of infrared light proportional to the concentration of gas present in the sample. When gas molecules absorb this infrared light, their temperature rises as the molecules increase in kinetic energy; this results in a pressure wave inside the acoustic cell. The audible pressure pulses corresponding to the light pulses are detected by a very sensitive microphone located inside the chamber (MSA, 2001) and are proportional to the concentration of CO₂ molecules.

3. Definitions of Terms and Acronyms

- | | | |
|-------|--------|--|
| 3.1. | AirDAC | Air Data Acquisition and Control program |
| 3.2. | CAB | Concentrated animal building |
| 3.3. | DAC | Data acquisition and control |
| 3.4. | FS | Full scale |
| 3.5. | GSS | Gas sampling system |
| 3.6. | MSA | Mine Safety Appliances, Inc. |
| 3.7. | NDIR | Non-dispersive infrared |
| 3.8. | OFIS | On-farm instrument shelter |
| 3.9. | QAPP | Quality Assurance Project Plan |
| 3.10. | PAAQL | Purdue Agricultural Air Quality Laboratory |
| 3.11. | RH | Relative humidity |

4. Health and Safety

- 4.1. Be careful when working with the electrical power connections.
- 4.2. Properly vent analyzer exhaust to avoid exposure to noxious gases in the sample.

5. Interferences

- 5.1. The following compounds or parameters interfere with the CO₂ reading:
 - 5.1.1. Methane (CH₄) causes a 4.2-ppm increase in the CO₂ reading per 1% CH₄ present. However, CH₄ concentrations above 0.05% are rare in CAB, so this interference is negligible.
 - 5.1.2. Carbon monoxide (CO), sulfur dioxide (SO₂), nitric oxide (NO), and ozone (O₃) each cause an approximate 0.5-ppm increase in the CO₂ reading per 1% present. However, concentrations of these gases are negligible in CAB compared to 1%.
 - 5.1.3. Temperature interferences are <0.4% FS per °C (<0.22% FS per °F). Since the analyzer and the GSS are kept inside the temperature controlled OFIS (82°F ± 8°F, SOP U1), the magnitude of this effect should be <2% of FS (<200 ppm).
 - 5.1.4. Humidity causes an increase of 0.05% FS per 1% RH, if the RH is below 20%. The interference is lower above 20% RH, and drops to near-zero above 40% if the instrument was calibrated with methane-spiked span gas. Use only methane-spiked span gases (Section 7.4) during calibration to avoid this interference. Zero gas does not need to contain methane.
 - 5.1.5. Pressure can cause an increase of 0.1% of FS per mBar. Since the analyzer draws from the analyzer manifold of the GSS, which is maintained at a constant pressure (slightly above atmospheric pressure), the pressure will not vary by as much as 1 mBar. This interference is thus negligible unless there is a malfunction in either the OFIS airflow or the GSS.
 - 5.1.6. N₂, O₂, Air, and He each have no photoacoustic effect, and therefore do not cause any interference.

6. Personnel Qualifications

- 6.1. Personnel should be trained in the use of the instrument before initiating the procedure. Training requires about three hours.
- 6.2. Read the entire instrument manual and this SOP before operating this analyzer.
- 6.3. Personnel should also be trained to interpret the analyzer output signal, converted concentration data, and correction of concentrations based on calibration.

7. Equipment and Supplies

- 7.1. Carbon dioxide analyzer (Model 3600, Mine Safety Appliances, Pittsburgh, PA)
- 7.2. Rack and rack mounts.
- 7.3. Keep the following spare parts on hand at each site:
 - 7.3.1. Replacement Solenoid Assembly. Part No. 100027655
 - 7.3.2. Pump assembly, Part No. 806808

- 7.3.3. Dampener, Part No. 459928
- 7.3.4. Finite filter Model IDN-6G
- 7.3.5. Flow switch assembly, Part No. 696606
- 7.4. Calibration gases (exact composition and specifications to be described in the QAPP for the particular project)
 - 7.4.1. CO₂ in N₂ with 2.5% CH₄
 - 7.4.2. Zero air
- 7.5. Appropriate regulators for calibration gas cylinders (SOP G2)

8. Procedure

- 8.1. Setup
 - 8.1.1. Check analyzer carefully to assure it is free of visible defects or damage.
 - 8.1.2. Securely mount the analyzer vertically on the instrument rack or wall.
 - 8.1.3. Connect the analog signal (4-20 mA) output of the analyzer at output terminals 7 (+) and 8 (-) (Fig. 2-3 in Model 3600 Manual) to the data acquisition system.
 - 8.1.4. Connect the GSS analyzer manifold to the analyzer inlet with ¼" ID Teflon tubing and connect the analyzer outlet to the air exhaust of the on-farm instrument shelter with ¼" ID PVC or other tubing.
 - 8.1.5. Power up the analyzer, turn on its internal pump, and allow the analyzer to warm up for at least 5 minutes before checking the concentration readings.
 - 8.1.6. Enter the analyzer's analog signal and measurement ranges into the AirDAC software so that concentration reading can be displayed, converted and recorded.
- 8.2. Gas Analyzer Calibration
 - 8.2.1. Reference gases
 - 8.2.1.1. Carbon dioxide (CO₂) in nitrogen (N₂) with 2.5% methane (CH₄) with CO₂ concentration and certification specified by the QAPP. The CH₄ acts to help the CO₂ molecules to convert their higher energy states from infrared photon absorption through collisions into thermal then acoustic energy. Also, CH₄ allows the Model 3600 to be properly calibrated without humidity present. For safety reasons, do not introduce a concentration above 2.5% CH₄ to the Model 3600.
 - 8.2.1.2. Zero air, which can be purchased as Continuous Emission Monitoring Zero Air.
 - 8.2.2. Follow SOPs G8 and G9 for calibration and precision checks. The number of points and the gas concentrations for the multipoint calibrations (SOP G8) and precision checks (SOP G9) are specified in the project's QAPP. Procedures for zero and span gases are the same, so "reference gas" is used herein to denote either gas.
 - 8.2.2.1. Zero/precision checks
 - 8.2.2.1.1. Documents responses to zero gas and a selected span gas.
 - 8.2.2.1.2. No analyzer adjustments are made or maintenance conducted during a check.
 - 8.2.2.2. Analyzer adjustments are conducted only when:
 - 8.2.2.2.1. Stabilized reading of the reference gas is outside the tolerances specified in Sections 10.3 (span gases) or 10.4 (zero gas).
 - 8.2.2.2.2. If analyzer is adjusted, then record the analyzer's responses to both zero and span gases in an as-found zero-precision check (SOP G8) immediately prior to adjusting the analyzer, even before the analyzer's first use. The as-found

responses are used to validate data since the previous calibration or previous zero/precision check.

- 8.2.3. Introduce the calibration reference (zero or span) gas and adjust the gas analyzer, if necessary. The procedure to introduce the reference gas is as follows:
 - 8.2.3.1. Record calibration date and start time in the lab notebook.
 - 8.2.3.2. Start flow of reference gas to the analyzer. See SOP G1 (Gas Sampling System) for various options of delivering the reference gas to the analyzer). The QAPP for a particular project will state which method is to be used.
 - 8.2.3.3. Record pertinent gas delivery parameters, e.g. pressure, flow, temperature, relative humidity (SOP G1).
 - 8.2.3.4. Wait 10 min for the display to stabilize and record the time and concentrations shown on the gas analyzer display by AirDAC in the field notebook.
 - 8.2.3.5. If, during a precision check (SOP G9), the stabilized reading of the reference gas is outside the tolerances specified in Sections 10.3 (span gases) or 10.4 (zero gas), then adjust the analyzer.
 - 8.2.3.6. If analyzer adjustment is needed (See Section 8.2.2.1) then:
 - 8.2.3.6.1. Open front door of the Model 3600.
 - 8.2.3.6.2. Adjust only the zero or span screw (Fig. 1b), as appropriate based on the gas being tested, until the concentration is within ± 50 ppm of the certified reference gas concentration.
 - 8.2.3.6.3. As per SOP G9, follow this adjustment with a multipoint calibration.
 - 8.2.3.7. Stop flow of reference gas to the gas analyzer.
 - 8.2.3.8. Record calibration date and end time in the lab notebook.
 - 8.2.3.9. Repeat the steps in Sections 8.2.3.2 through 8.2.3.6 for each reference gas.
 - 8.2.3.10. Return analyzer to gas sampling mode.
 - 8.2.3.11. Return AirDAC to automatic sampling.
- 8.3. Troubleshooting
 - 8.3.1. Troubleshooting guidelines are in Table 5-1 of the instrument manual.
 - 8.3.1.1. Check wiring and power connections if there is no signal or power.
 - 8.3.1.2. Check pump if there is no flow or concentrations too low.
 - 8.3.1.3. Check filter and replace if dirty.
 - 8.3.1.4. Check flow path integrity if reference gas concentration cannot be reached or sample flow rate is low.
 - 8.3.1.5. Clean or replace leaking solenoids. Solenoids (Fig. 2) that cannot close properly will cause a leakage into the acoustic cell, resulting in slow response and inability to reach the span gas value.
- 8.4. Data acquisition, calculations & data reduction requirements
 - 8.4.1. Monitor a 4-20 mA analog output using AirDAC.
 - 8.4.2. Report data with 3 significant digits.
 - 8.4.3. Follow SOP B4 to correct gas concentrations for calibration and air density.
- 8.5. Computer hardware & software
 - 8.5.1. AirDAC (SOP B2)
 - 8.5.2. ARDIS (SOP B3)
 - 8.5.3. CAPECAB (SOP B6)

9. Data and Records Management

- 9.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record with a record book designated for the method, and include copies of the electronic record.
- 9.2. Manage data according to SOP B5 “Management of Barn Data”.
- 9.3. Document all data and information (e.g., sample collection method used) on field data sheets, and within site logbooks with permanent ink, or in electronic field notes.

10. Quality Control and Quality Assurance

- 10.1. Conduct gas analyzer checks at a frequency specified by the QAPP. A typical frequency is weekly, but some QAPP may require a different frequency. This will include zero gas and one or more span concentrations.
- 10.2. Report QC data using a control chart in an Excel spreadsheet (SOP Q1).
- 10.3. Conduct gas analyzer multipoint calibration when the precision check of the gas analyzer with a span gas drifts more than 5% from the established baseline value.
- 10.4. Conduct gas analyzer calibrations when the precision check reading with a zero gas drifts more than 2% of full scale from the established zero baseline.
- 10.5. The calibration gas should be certified using EPA protocol (1% certification).
- 10.6. Check the response time every month, or at a frequency specified by the QAPP. The manual indicates that the instrument response to a step-change is 97% in 12 s.
- 10.7. Measure the gas analyzer inlet flow rate every six months.
- 10.8. Perform leak check in sample line and flow components and fittings when the equipment is installed, and then every six months. Plug the inlet and verify that the exhaust flow is zero using a flow meter.
- 10.9. Monitor sampling manifold temperature, relative humidity and pressure.

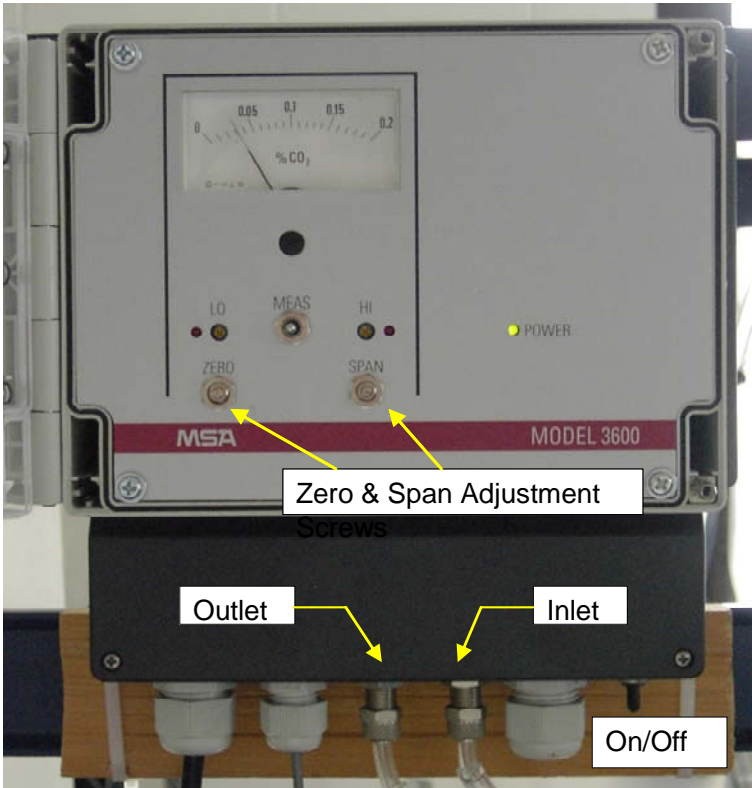
11. References

- 11.1. Mine Safety Appliances. 1993. MSA Model 3600 Infrared Gas Monitor Instruction Manual. (L) Rev. 2. [media.msanet.com/NA/USA/PermanentInstruments/HVACMonitors/Model3600InfraredGasMonitor/800031Rev2.pdf]. Accessed 11/7/05.
- 11.2. Roczko, A. 2001. MSA Photoacoustic Infrared Technology for Detection of Refrigerant Gases. [media.msanet.com/NA/USA/PermanentInstruments/HVACMonitors/ChillgardLERefrigerantMonitor/PhotoacousticRefrigerantWhitePaper.pdf] Accessed 11/7/05.
- 11.3. SOP B2. 2006. Data Acquisition and Control Software (AirDAC). Standard Operating Procedure B2. Purdue Ag Air Quality Lab.
- 11.4. SOP B3. 2006. Air Data Pre-Processing Software. Standard Operating Procedure B3. Purdue Ag Air Quality Lab.
- 11.5. SOP B4. 2006. Calculation and Reporting of Air Emissions from Barns. Standard Operating Procedure B4. Purdue Ag Air Quality Lab.

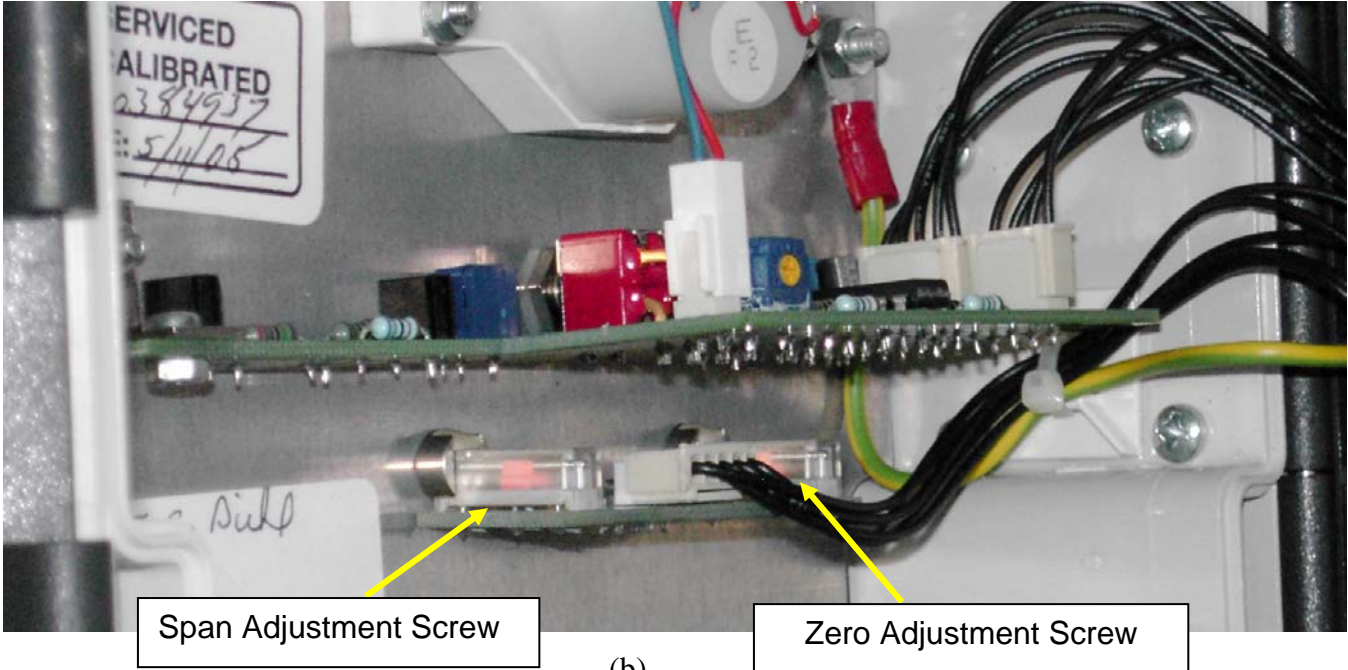
- 11.6. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 11.7. SOP B6. 2006. Data Processing Software (CAPECAB). Standard Operating Procedure B6. Purdue Ag Air Quality Lab.
- 11.8. SOP G1. 2006. The PAAQL Gas Sampling System. Standard Operating Procedure G1. Purdue Ag Air Quality Lab.
- 11.9. SOP G2. 2006. Compressed Gas Cylinders. Standard Operating Procedure G2. Purdue Ag Air Quality Lab.
- 11.10. SOP G8. 2006. Multi-Point Calibration of Gas Analyzers. Standard Operating Procedure G8. Purdue Ag Air Quality Lab.
- 11.11. SOP G9. 2006. Precision Checks of Gas Analyzers. Standard Operating Procedure G9. Purdue Ag Air Quality Lab.
- 11.12. SOP Q1. 2006. Use of Control Charts for Performance Monitoring of Gas Analyzers and Analytical Instruments. Standard Operating Procedure Q1. Purdue Ag Air Quality Lab.
- 11.13. SOP U1. 2006. On-Farm Instrument Shelters for Barn Sources. Standard Operating Procedure U1. Purdue Ag Air Quality Lab.

12. Contact Information

- 12.1. Distributor: J&M Instrument Co., 7050 Greybudd Dr., Indianapolis, IN, 317-290-9127
- 12.2. Manufacturer Technical Support: 1-888-421-8324
<http://www.msanorthamerica.com/customerservice.html>.
- 12.3. PAAQL: odor@purdue.edu, heber@purdue.edu, <http://www.AgAirQuality.com>.



(a)



(b)

**Figure 1. Location of zero and span screws from outside (a) and inside (b) the analyzer.
Note: The analyzer in this picture has a 2,000 ppm range.**

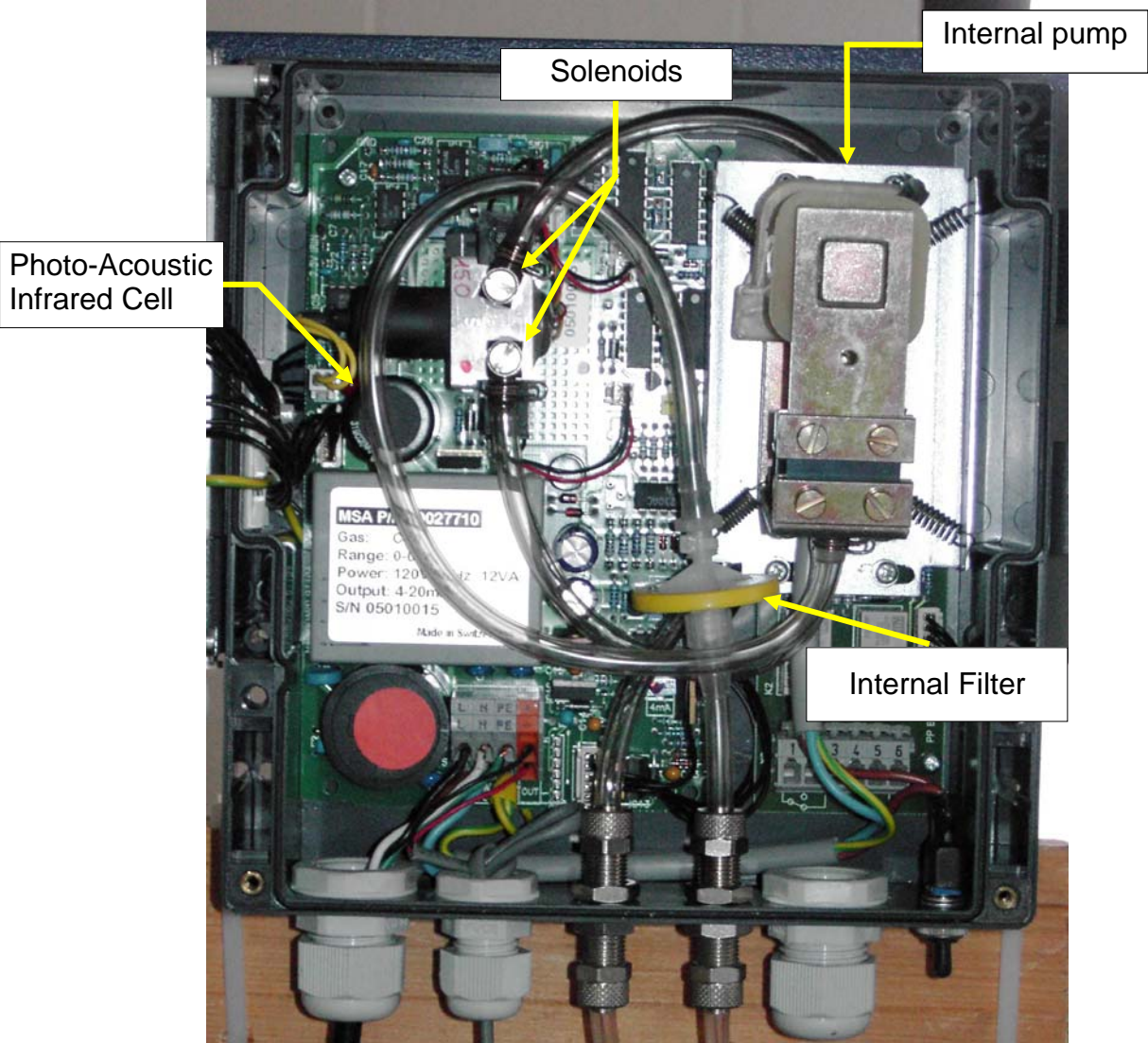


Figure 2. Location of key components of the analyzer.

**MEASUREMENT OF AMMONIA (NH₃) USING THE TEC
MODEL 17C CHEMILUMINESCENCE ANALYZER**

Standard Operating Procedure (SOP) G4

**MEASUREMENT OF AMMONIA (NH₃) USING THE TEC
MODEL 17C CHEMILUMINESCENCE ANALYZER
Standard Operating Procedure (SOP) G4**

Prepared by

Bill W. Bogan, Juan Carlos Ramirez, and Claude A. Diehl

Reviewed by

Albert J. Heber

Effective Date: November 6, 2006

**PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)
Agricultural and Biological Engineering, Purdue University
West Lafayette, IN 47907**

Table of Contents

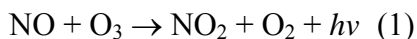
	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions of Terms and Acronyms	3
4. Health and Safety	4
5. Cautions	4
6. Interferences	4
7. Personnel Qualifications.....	5
8. Equipment and Supplies	5
9. Procedures	5
10. Data and Records Management	9
11. Quality Control and Quality Assurance	9
12. References.....	10
13. Contact Information	10

1. Scope and Applicability

- 1.1. The purpose of this procedure is to present the methodology and instrumentation used to measure automatically and sequentially concentrations of ammonia (NH₃) emissions at animal feeding operations (AFO), specifically at the barns.
- 1.2. This procedure applies only to the Thermo Electron Model 17C Chemiluminescence NH₃ Analyzer.
- 1.3. The chemiluminescence method is useful for measuring NH₃ in barn air due to its low minimum detection limit, short response time and wide measurement range.
 - 1.3.1. The detection limit of the method is 1 ppb.
 - 1.3.2. The 90% response time to a step change in NH₃ concentration is 120 sec.
- 1.4. The standard range is from 0 to 100,000 ppb (0 to 100 ppm); adjusting the photomultiplier tube voltage can extend this range to 0 - 200 ppm.
- 1.5. The stated precision of the TEC 17C is 0.5% of full scale.

2. Summary of Method

The chemiluminescence NH₃ analyzer (Model 17C, Thermal Environmental Instruments (TEC), Franklin, MA) is a combination NH₃ converter and an NO-NO₂-NO_x analyzer (See Fig. 1). The air sample containing ammonia is pulled into the analyzer using an external vacuum pump. The ammonia in the air sample is oxidized to nitric oxide (NO) with a stainless steel catalytic converter at 825°C. The NO is further oxidized with ozone (O₃) in the analyzer's reaction chamber producing nitrogen dioxide (NO₂) molecules in an excited state that emit radiation as they return to a lower energy state (Equation 1). The intensity of the emitted radiation is proportional to the concentration of NO.



where O₂ is oxygen and $h\nu$ represents photons, particles of light energy, or radiation energy that is generated by NO₂ in an excited state returning to the ground state. The emitted radiation is detected by a photomultiplier tube (PMT), which in turn generates an electronic signal that is processed into a gas concentration reading. Sample air is drawn at a flow rate of 0.6 L/min from the converter into the NH₃ analyzer through a particulate filter, a glass capillary, and a solenoid valve. The solenoid valve routes the sample either directly into the reaction chamber (NO mode), through the molybdenum converter at 325°C and the reaction chamber (NO_x mode), or through the stainless steel catalytic converter and the reaction chamber (N_t mode). Ammonia concentration is calculated based on the difference between the readings obtained by the N_t and NO_x modes. The response time of the 17C is decreased if operated only in the N_t mode.

3. Definitions of Terms and Acronyms

- 3.1. DAC Data acquisition and control
- 3.2. GSS Gas sampling system
- 3.3. MSDS Material safety data sheet
- 3.4. OFIS On-farm instrument shelter
- 3.5. PAAQL Purdue Agricultural Air Quality Laboratory

- 3.6. PMT Photomultiplier tube
- 3.7. QAPP Quality Assurance Project Plan
- 3.8. RH Relative humidity
- 3.9. TEC Thermo Electron Corporation

4. Health and Safety

- 4.1. The exhaust stream leaving the reaction chamber will have significant concentrations of ozone and oxides of nitrogen. Incorrect connections between the analyzer and the vacuum pump, and between the vacuum pump and the exhaust could cause a significant health and safety hazard.
 - 4.1.1. Ozone at 0.3 ppm for 30 min can cause eye and skin irritation, headache, nausea, and changes in pulmonary function (lung impairment and breathing difficulty). Death may occur from prolonged exposure at 2 ppm or short exposures (1 to 4 h) at 10 ppm (See the MSDS for Ozone located in the OFIS).
 - 4.1.2. Oxides of nitrogen (nitric oxide (NO) and nitrogen dioxide (NO₂)) can cause eye and skin irritation, headache, nausea, and changes in pulmonary function (lung impairment and breathing difficulty) within 72 h of exposure (See the MSDSs for NO and NO₂ located in the OFIS).
- 4.2. Some of the internal components of the 17C work at high temperatures. Let the 17C cool down before touching or accessing those parts.
- 4.3. Be careful when working with the electrical power connection.

5. Cautions

- 5.1. Monitor the NH₃ conversion efficiency (NH₃ coefficient, See Section 9.4.3) of the 17C. A conversion efficiency < 80% (coefficient < 0.8) will cause the ammonia scrubbers to become clogged more rapidly, requiring extra expense and down time while the scrubbers are changed. If the coefficient drops below 0.8, see Section 9.4.3.
- 5.2. Failure of the permeation dryer due to contamination of the inner tubing (Section 11.8.2) can lead to a non-linear response.
 - 5.2.1. Use antistatic wrist strap or discharge your body's electrostatic charge to a grounded metallic frame before touching any internal components of the 17C to avoid damage of the electronic components by electrostatic discharge. For further information, see Chapter 7 ("Servicing") of the 17C manual.

6. Interferences

- 6.1. Nitrogen dioxide (NO₂) at approximately 100 ppb causes a small (3 to 6%) negative response (i.e. an underestimate of NH₃ concentrations) (USEPA/Battelle 2004a & b).
 - 6.1.1. NO₂ levels in barns are expected to be negligible.
- 6.2. Diethylamine at approximately 100 ppb causes a response approximately 50% of that seen with NH₃ at the same concentration (i.e. an overestimate of NH₃ concentrations equal to 50% of the diethylamine concentration) (USEPA/Battelle 2004a & b). Other alkylamines which are likely to be present at livestock sites (e.g. methylamine, dimethylamine, ethylamine) may present similar interferences.
 - 6.2.1. Amine levels in barns are expected to be negligible compared to those of NH₃.

7. Personnel Qualifications

- 7.1. Personnel must be trained in the use of the 17C before initiating the procedure. Training requires about three hours.
- 7.2. Each analyst must read and understand the entire 17C manual and this SOP before operating the 17C.
- 7.3. Personnel must be able to interpret the analyzer output signal, converted concentration data, and correction of concentrations based on calibration.

8. Equipment and Supplies

- 8.1. Chemiluminescence NH₃ analyzer (Model 17C, Thermo Electron Corporation, Franklin, MA)
- 8.2. Rack and rack mounts
- 8.3. Keep the following spare parts at each remote site:
 - 8.3.1. Ammonia scrubbers, Part Nos. 10157, 10158, 10159
 - 8.3.2. Capillary – 10 mil long, Part No. 4121
 - 8.3.3. Ozone capillary, Part No. 4119
 - 8.3.4. O-ring for capillary, Part No. 4800
 - 8.3.5. Fuse - 5 amp, Part No. 4523
 - 8.3.6. Fuse - 3 amp slo-blo, Part No. 4510
- 8.4. Keep the following spare parts in a central stock at PAAQL:
 - 8.4.1. Stainless steel converter cartridge, Part No. 10155
 - 8.4.2. Solenoid valve, Part No. 8119
 - 8.4.3. Molybdenum (Moly) converter cartridge, Part No. 9269
 - 8.4.4. Thermocouple - Moly, Part No. 9204
 - 8.4.5. Thermocouple, Part No. 10170, or equivalent
 - 8.4.6. Bandheater - Moly (110 volt), Part No. 9261
 - 8.4.7. Pump, Part No. 9456, or equivalent
 - 8.4.8. Pump repair kit, Part No. 9464
 - 8.4.9. Permeation dryer assembly, Part No. 60800
- 8.5. Calibration gases: Ammonia, nitric oxide, zero gas (exact specifications and compositions to be defined in the QAPP)

9. Procedures

- 9.1. Gas Analyzer Setup
 - 9.1.1. Carefully check analyzer and converter for visible defects or damage.
 - 9.1.2. Securely mount the analyzer and converter in the instrument rack.
 - 9.1.3. Using 1/4" OD Teflon tubing, connect "NO OUT", "NO_x OUT", and "N_t OUT" bulkheads on the rear panel of the converter to "NO IN", "NO_x IN", and "N_t IN" bulkheads, respectively, on the analyzer rear panel (Fig. 1).
 - 9.1.4. Connect the analyzer and converter with the communication cable.
 - 9.1.5. Connect the GSS analyzer manifold to the "SAMPLE" bulkhead on the rear panel of the converter, using a 1/4" OD (1/8" ID) Teflon tube less than 10' long.
 - 9.1.5.1. Allow enough slack to permit movement of the instrument rack.
 - 9.1.5.2. Use tubing that is clean, dry, and uncontaminated.

- 9.1.6. Connect the vacuum inlet (stainless steel fitting) of the twin-headed vacuum pump to the analyzer's "CHAMBER" bulkhead. See Section 4.1.
- 9.1.7. Connect the vacuum pump exhaust port to the OFIS air exhaust with 1/4" OD (1/8" ID) PVC tube that is less than 10' long.
- 9.1.8. Connect the analyzer's analog output port to the data acquisition system using shielded cable. See Table 2-1 of the 17C manual for recommended cable types.
 - 9.1.8.1. Properly ground the shielded analog output cable.
 - 9.1.8.1.1 Remove about 1.8 cm of insulation from the end of the cable.
 - 9.1.8.1.2 Fold back the shielding. If necessary, use electrical tape or shrink tubing to hold shielding in place. Expose a minimum of 1.5 cm of shielding.
 - 9.1.8.1.3 Strip each signal wire.
 - 9.1.8.1.4 Connect shielded cable to the slots on the 8-position header, as shown in Figure 2-5 of the 17C manual.
- 9.1.9. Plug in the sample pump, turn on the analyzer, converter and ozonator, and run Model 17C overnight before calibrating.
- 9.1.10. Set operating ranges and averaging times to appropriate settings (Table 1). See Chapter 3 of the 17C manual for more details about 17C parameters. Fig. 2 in the manual includes a menu flow chart.

Table 1. Appropriate settings for the TEC 17C.

Parameter	Range
Internal temperature, °C	15 - 45
Chamber temperature, °C	47 - 51
Capillary temperature, °C	48 - 50
Cooler temperature, °C	-10 to -1
NO ₂ converter temperature, °C	325 - 350
NH ₃ converter temperature, °C	775 - 825
Pressure, mm Hg	50 - 300
Sample flow, L/min	0.30 - 0.90
Ozonator flow, L/min	0.05 - 0.25

- 9.1.11. Set averaging time to 10 s for fastest dynamic response.
 - 9.1.12. Set to "Auto" mode using the front panel (Fig. 2).
 - 9.1.13. Set to a single range mode by turning off option switches 4 and 5.
 - 9.1.14. Select and enter the values for NO, NO₂, NO_x, Nt, and NH₃ ranges. The ranges of the gases depend on the time of year and type of livestock, as shown in Table 2.
 - 9.1.15. Enter the analog signal and measurement ranges into the AirDAC software so that concentrations can be displayed, converted and recorded.
- 9.2. Gas Analyzer Calibration
- 9.2.1. Reference calibration gases
 - 9.2.1.1. Nitric oxide (NO) in N₂: EPA Protocol ($\pm 1\%$ analytical accuracy) if NO is primary data. Exact composition specified in the QAPP.
 - 9.2.1.2. NH₃ in air: Oxygen is required to convert NH₃ to NO. The ammonia standard should have a analytical accuracy of $\pm 1\%$. Exact composition specified in the QAPP.
 - 9.2.1.3. Zero air, typically referred to as Continuous Emission Monitoring Zero Air.

Table 2. Expected range (ppm) for each analyte, as a function of livestock species and season.

Gas	Swine		Poultry		Dairy	
	Expected Maximum Concentration					
	Summer	Winter	Summer	Winter	Summer	Winter
NH ₃	20	50	50	200	10	30
NO	< 1	< 1	< 1	< 1	< 1	< 1
NO ₂	< 1	< 1	< 1	< 1	< 1	< 1
NO _x	< 1	< 1	< 1	< 1	< 1	< 1
N _t	20	50	50	200	10	30
	Proper Range Setting for 17C					
	Summer	Winter	Summer	Winter	Summer	Winter
NH ₃	20	50	50	200	10	30
NO	1	1	1	1	1	1
NO ₂	1	1	1	1	1	1
NO _x	1	1	1	1	1	1
N _t	20	50	50	200	10	30

- 9.2.2. Calibrate the 17C following SOPs G8 (multipoint calibrations) and G9 (zero/precision checks).
- 9.2.2.1. Set the averaging time to 60 s for calibration.
- 9.2.2.2. Adjustment of the analyzer. A precision check is to be performed before any adjustment of the analyzer. The number of points and gas concentrations for multipoint calibrations and precision checks are specified in the Quality Objectives of the QAPP.
- 9.2.2.3. Record calibration or precision check date and time in field notes (Table 4).
- 9.2.2.4. Record 17C pressure, sample flow, and ozone flow at each point (Table 3).
- 9.2.2.5. Start flow of reference gas to the analyzer. See SOP G1 for delivery options.
- 9.2.2.6. Record pertinent gas delivery parameters (Concentration and flow rate (SOP G11))
- 9.2.2.7. Wait a minimum of 10 min for the display to stabilize.
- 9.2.2.8. Record the time and concentrations shown on the 17C display, and by AirDAC, in the field notebook.
- 9.2.2.9. If the stabilized concentration during a precision check deviates from zero by 2% of full scale and / or the baseline concentration by 10% or more, adjust the gas analyzer calibration as follows:
- 9.2.2.9.1 Zero adjustment
- 9.2.2.9.1.1 Choose “Calibration” from the Main Menu, and choose “Calibrate Zero” from the Calibration Menu.
- 9.2.2.9.1.2 Press “ENTER” to set the NO, NO_x, and N_t readings to zero.
- 9.2.2.9.1.3 Press “RUN” to return to the Run Screen.
- 9.2.2.9.2 Precision adjustment
- 9.2.2.9.2.1 Choose “Calibration” from the Main Menu, and choose “Calibrate NO, NO_x, or, NH₃” from the Calibration Menu.
- 9.2.2.9.2.2 The first line of the Calibrate NO, NO_x, or, NH₃ screen displays the current NO, NO_x, or, NH₃ reading.

- 9.2.2.9.2.3 Enter the actual NO, NO_x, or, NH₃ calibration gas concentration on the third line of the display. Position the cursor using the ← and → buttons. Adjust each digit in the reading using the ↑ and ↓ buttons.
- 9.2.2.9.2.4 Press “ENTER” when the concentration is correct.
- 9.2.2.9.2.5 Stop gas flow and record the time.
- 9.2.3. Repeat the steps in Section 9.2.2 for each reference gas.
- 9.2.4. Set the analyzer’s averaging time back to 10 s.
- 9.2.5. Return analyzer to gas sampling mode. This includes setting AirDAC to auto sample.
- 9.3. Gas Analyzer Precision Checks
 - 9.3.1. Repeat steps 9.2.2.3 through 9.2.5, but skip adjustment steps (Sections 9.2.2.9. through 9.2.2.9.2.5) and 9.2.4.
 - 9.3.2. Check soon after any repair or maintenance to verify that problem has been solved.
 - 9.3.3. Use QAPP-specified frequency, replications and reference gas concentrations.
- 9.4. Adjustment of the measurement range of the analyzer
 - 9.4.1. Run a precision check (SOP G9)
 - 9.4.2. Introduce NH₃ to the GSS using the Environics diluter
 - 9.4.2.1. Use 180 ppm of NH₃ to set the measurement range for NH₃ to 200 ppm
 - 9.4.2.2. Use 90 ppm of NH₃ to set the measurement range for NH₃ to 100 ppm
 - 9.4.3. Wait until measurement (reading) stabilizes or after 10 min.
 - 9.4.4. Remove the TEC 17C NH₃ analyzer cover.
 - 9.4.5. Locate the blue PMT high voltage power supply (the only blue box in the analyzer).
 - 9.4.5.1. Using a small standard screwdriver, adjust adjustment screw on the top of the box.
 - 9.4.5.1.1 When introducing NH₃ at 180 ppm, turn the screw counterclockwise until the analyzer’s reading is stabilized at 90 ppm.
 - 9.4.5.1.2 When introducing NH₃ at 90 ppm, turn the screw clockwise until the analyzer’s reading is stabilized at 90 ppm.
 - 9.4.6. Place the TEC 17C NH₃ analyzer cover back on the analyzer.
 - 9.4.7. Perform the following after adjusting the measurement range of the analyzer:
 - 9.4.7.1. Precision Check (SOP G9)
 - 9.4.7.2. Multipoint Calibration (SOP G8)
 - 9.4.7.3. Precision Check to establish a new baseline for the control chart (SOP Q1)
- 9.5. Maintenance
 - 9.5.1. Clean the dust filters on the back of the analyzer and converter on a regular basis. This can range from weekly to monthly, depending on the dust level in the OFIS.
 - 9.5.2. Track the operating pressure of the 17C in its control chart (See SOP Q1). Replace the ammonia scrubbers when frequent analyzer adjustments are required (i.e. when the unit repeatedly drifts outside the 10% tolerance limit – usually the lower bound due to reduced sensitivity – around the certified concentration of the precision check gas), and when pressure is increasing (Fig. 3).
 - 9.5.2.1. These two factors indicate salt buildup within the analyzer’s stainless steel cross and/or exhaust tubing (Fig. 4).
 - 9.5.2.1.1 Clean the stainless steel cross and/or exhaust tubing as necessary.
 - 9.5.2.1.2 Conduct a zero & precision span check before and after cleaning.
 - 9.5.2.2. Replace scrubbers more frequently when measuring high ammonia concentrations.

- 9.5.3. Check the NH_3 coefficient (found in the “CALIBRATION FACTORS” Menu) on a weekly basis, and record in the electronic field notes. See Section 5.1.1 if the NH_3 coefficient is < 0.8 . If the coefficient drops below 0.8:
 - 9.5.3.1. Ensure that there are no leaks in the sampling system that may be resulting in loss of NH_3 .
 - 9.5.3.2. If no leaks or other problems can be found with the sampling system, contact the manufacturer to troubleshoot the N_1 converter.
- 9.6. Troubleshooting
 - 9.6.1. Troubleshooting guidelines are in Chapter 6 of the 17C manual.
 - 9.6.1.1. Check wiring and power connections and fuse if there is no signal or power.
 - 9.6.1.2. Check pump if there is no flow or no exhaust, or if concentrations are too low.
 - 9.6.1.3. Check filter and replace if dirty.
 - 9.6.1.4. Check flow path integrity if span concentration is unattained, or if flow rate is low.
 - 9.6.1.5. If the cooler will not hold its set temperature, check the dust filters, and make sure that the temperature inside the OFIS is not too high ($> 95^\circ\text{F}$).
- 9.7. Data acquisition, calculations & data reduction requirements
 - 9.7.1. Monitor a 0-10 VDC analog output signal using AirDAC.
 - 9.7.2. Report all data with 3 significant digits.
 - 9.7.3. Follow SOP B4 to correct gas concentrations for calibration and air density.
- 9.8. Computer hardware & software
 - 9.8.1. AirDAC (SOP B2)
 - 9.8.2. ARDIS (SOP B3)
 - 9.8.3. CAPECAB (SOP B6)

10. Data and Records Management

- 10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
- 10.2. Manage all data according to SOP B5.
- 10.3. Document all data and information (e.g., sample collection method used) on field data sheets, and within site logbooks with permanent ink, or in electronic field notes.

11. Quality Control and Quality Assurance

- 11.1. Verify compositions of all calibration gases by FTIR, according to SOP G12.
- 11.2. Conduct zero and one or more precision checks at the QAPP-specified frequency.
- 11.3. Conduct a gas analyzer calibration every 180 d (six months), or at the QAPP-specified frequency.
 - 11.3.1. The precision of the replicate points in this calibration should be $\pm 5\%$, unless another value is specified in the QAPP.
 - 11.3.2. The correlation coefficient (R^2) of the linear regression for the calibration should be $\geq 98\%$, unless another value is specified in the QAPP.
- 11.4. Conduct a gas analyzer calibration whenever the zero check is $> \pm 5\%$ of full scale different than the baseline zero, or at a limit specified by the QAPP.
- 11.5. Conduct a gas analyzer calibration whenever the precision check deviates from the baseline concentration by more than 10%, unless the QAPP indicates otherwise.

- 11.6. Perform a “bag test” with a known concentration of NH₃ (as described in SOP G1) every 60 days, or at the frequency designated in the QAPP.
- 11.7. Check response time every six months.
- 11.8. Perform preventative maintenance according to the schedule provided in Chapter 5 of the 17C manual, as follows:
 - 11.8.1. Check the inner tubing of the permeation dryer once per year to see if a dark yellow to brownish discoloration is present, change the permeation dryer.
 - 11.8.2. Check converter and analyzer capillaries for particulate deposits (pg. 5-2), and replace as necessary.
 - 11.8.3. Inspect and clean photomultiplier tube cooler fins (pg. 5-3) every 6 mos (minimum).

12. References

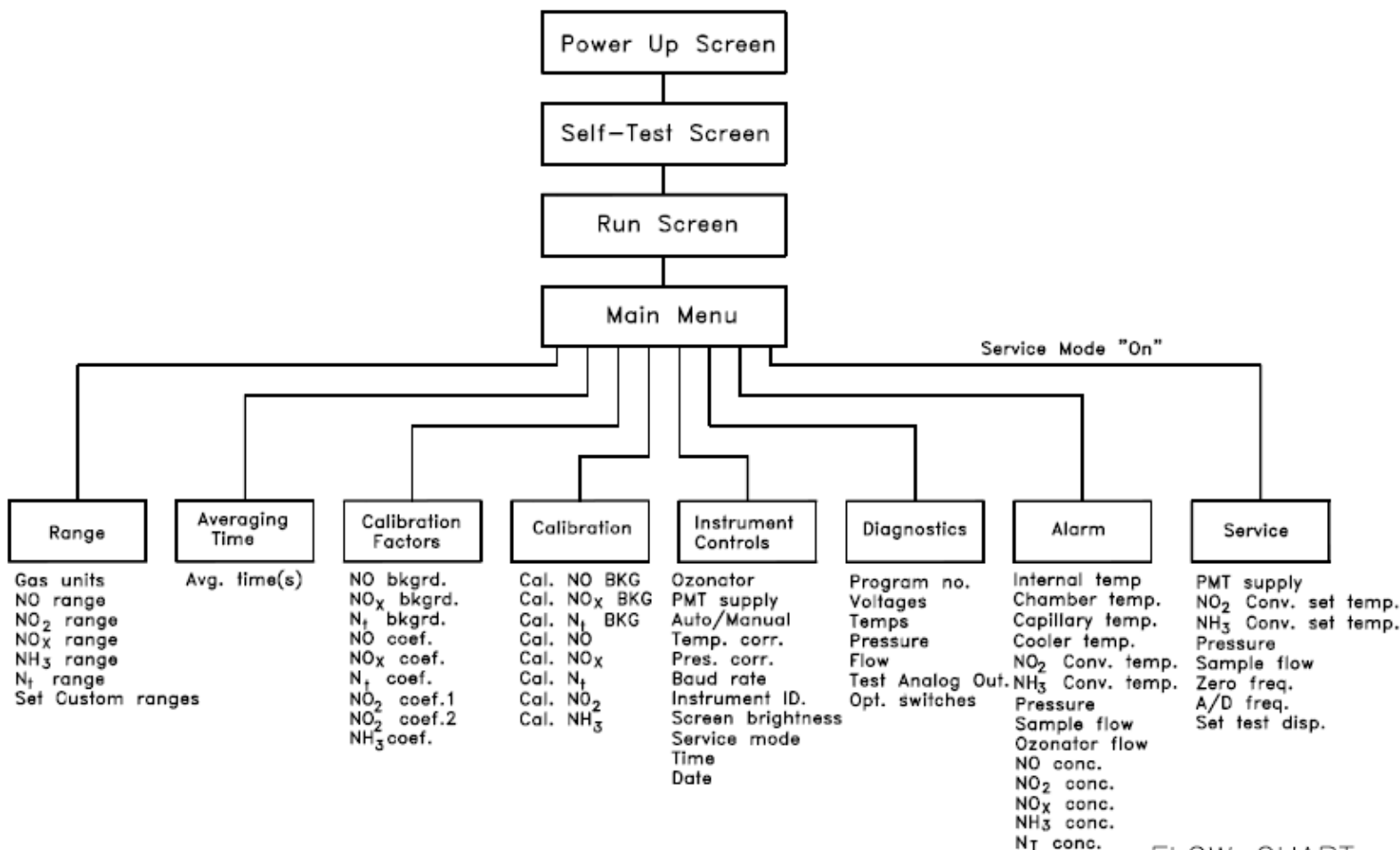
- 12.1. 17C Manual. TEC Model 17C Chemiluminescence NH₃ Analyzer Instruction Manual. Thermo Electron Corporation, Franklin, MA.
- 12.2. USEPA/Battelle. 2004a. ETV Joint Verification Statement. Model 17C Ammonia Analyzer. [http://www.epa.gov/etv/pdfs/vrvs/01_vs_thermo.pdf]. Accessed 1/27/2006.
- 12.3. USEPA/Battelle. 2004b. ETV Joint Verification Report. Model 17C Ammonia Analyzer. [http://www.epa.gov/etv/pdfs/vrvs/01_vr_thermo.pdf]. Accessed 1/27/2006.
- 12.4. SOP B2. 2006. Data Acquisition and Control Software (AirDAC). Standard Operating Procedure B2. Purdue Ag Air Quality Lab.
- 12.5. SOP B3. 2006. Air Data Pre-Processing Software. Standard Operating Procedure B3. Purdue Ag Air Quality Lab.
- 12.6. SOP B4. 2006. Calculation and Reporting of Air Emissions from Barns. Standard Operating Procedure B4. Purdue Ag Air Quality Lab.
- 12.7. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 12.8. SOP B6. 2006. Data Processing Software (CAPECAB). Standard Operating Procedure B6. Purdue Ag Air Quality Lab.
- 12.9. SOP G1. 2006. The PAAQL Gas Sampling System. Standard Operating Procedure G1. Purdue Ag Air Quality Lab.
- 12.10. SOP G2. 2006. Compressed Gas Cylinders. Standard Operating Procedure G2. Purdue Ag Air Quality Lab.
- 12.11. SOP G8. 2006. Multi-Point Calibration of Gas Analyzers. Standard Operating Procedure G8. Purdue Ag Air Quality Lab.
- 12.12. SOP G9. 2006. Zero and Precision Checks of Gas Analyzers. Standard Operating Procedure G9. Purdue Ag Air Quality Lab.
- 12.13. SOP G11. 2006. Operation of the Environics[®] Computerized Gas Dilution System. Standard Operating Procedure G11. Purdue Ag Air Quality Lab.
- 12.14. SOP Q1. 2006. Use of Control Charts for Performance Monitoring of Gas Analyzers and Analytical Instruments. Standard Operating Procedure Q1. Purdue Ag Air Quality Lab.

13. Contact Information

- 13.1. Manufacturer: Thermo Environmental Corporation (www.thermo.com), 8 West Forge Parkway, Franklin, MA 02038, Technical Support: 1-508-520-0430
- 13.2. PAAQL: odor@purdue.edu, heber@purdue.edu, <http://www.AgAirQuality.com>.



Figure 1: The front and back of the TEC 17C Ammonia Analyzer.



FLOW CHART

Figure 2: The Menu Flow Chart for the TEC 17C.

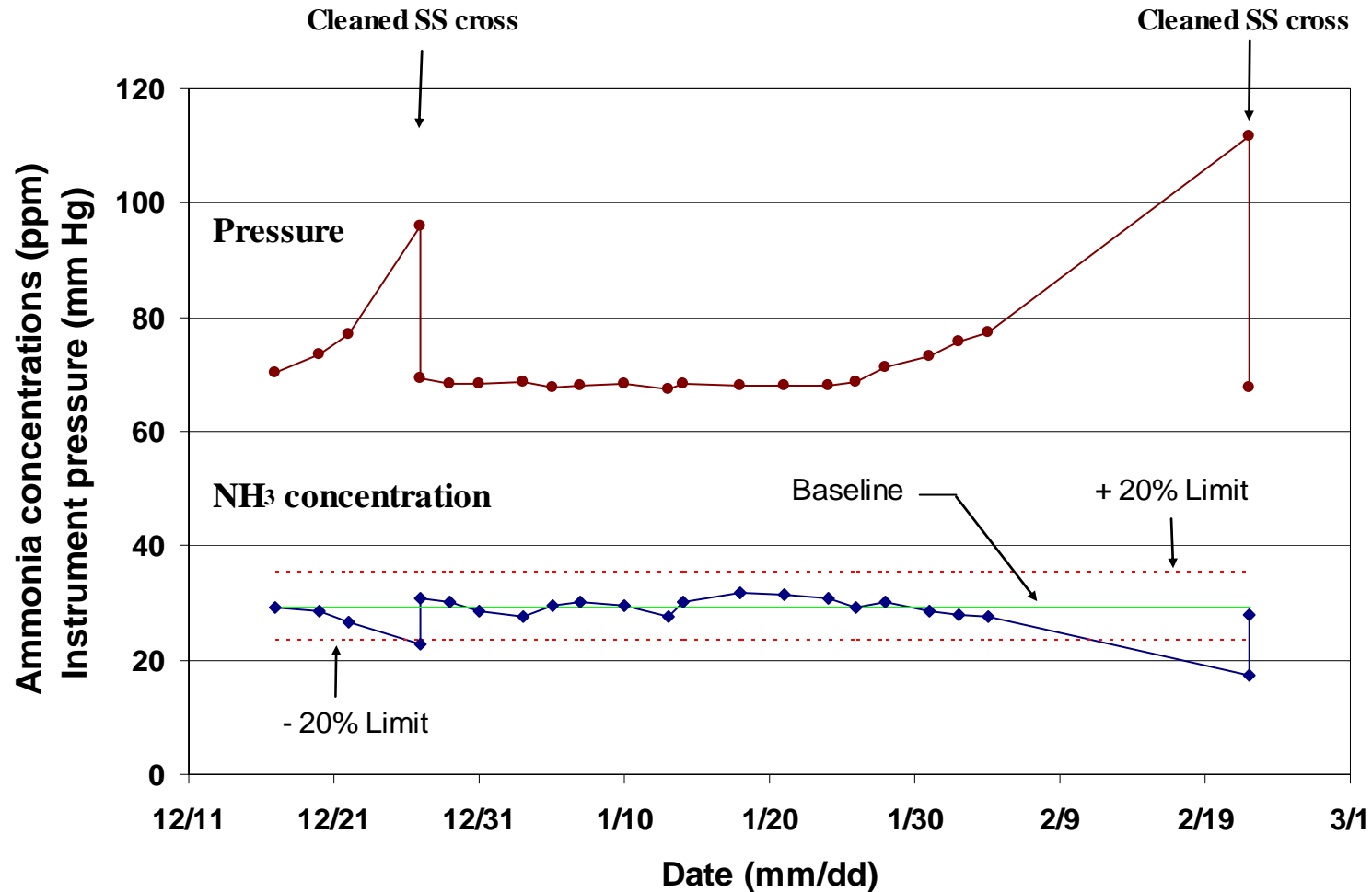


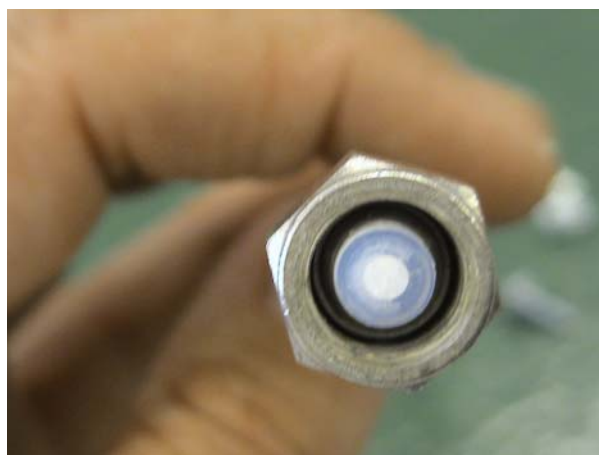
Figure 3. Graph, adapted from a TEC 17C control chart, showing NH₃ concentrations recorded during precision checks and operating pressure. Sharp increases in pressure, corresponding to decreased sensitivity (out-of-bounds low readings) indicate a need to clean out salt deposits (in this case, from the stainless steel cross).



a



b



c

Figure 4. Location of the 17C stainless steel cross (a), and buildup of ammonia salts in the stainless steel cross (b) and exhaust tubing (c).

**MEASUREMENT OF HYDROGEN SULFIDE (H₂S) WITH THE
THERMO ELECTRON CORPORATION MODEL 450I
PULSED-FLUORESCENCE ANALYZER**

Standard Operating Procedure (SOP) G5

**MEASUREMENT OF HYDROGEN SULFIDE (H₂S) WITH THE
THERMO ELECTRON CORPORATION MODEL 450I
PULSED-FLUORESCENCE ANALYZER
Standard Operating Procedure (SOP) G5**

Prepared by

Claude A. Diehl, Bill W. Bogan, Richard H. Grant and Matt T. Boehm

Reviewed by

Ji-Qin Ni and Albert J. Heber

Effective Date: November 6, 2006

Rev. 1.0 Effective Date: March 17, 2008

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions of Terms and Acronyms	4
4. Health and Safety	4
5. Interferences	5
6. Personnel Qualifications.....	5
7. Equipment and Supplies	5
8. Procedure.....	6
9. Data and Records Management	Error! Bookmark not defined.
10. Quality Control and Quality Assurance	16
11. References	17
12. Contact Information	17

1. Scope and Applicability

- 1.1. The purpose of this procedure is to present the methodology and instrumentation used to automatically and sequentially measure hydrogen sulfide (H₂S) emissions from barns, other buildings or open sources at confined animal feeding operations (CAFO).
- 1.2. This procedure applies only to the Model 450I Pulsed Fluorescence H₂S/SO₂ Analyzer (Thermo Electron Corporation, Franklin, MA).
- 1.3. The pulsed fluorescence method is useful for measuring hydrogen sulfide (H₂S) in air, due to its suitable detection limit, response time, and measurement range.
 - 1.3.1. The detection limit of the method is:
 - 1.3.1.1. 6 ppb (when using a 10-s averaging time)
 - 1.3.1.2. 2 ppb (60-s averaging time)
 - 1.3.1.3. 1.5 ppb (300-s averaging time)
 - 1.3.2. The 90% response time to a step change in H₂S concentration is:
 - 1.3.2.1. 80 s (10-s averaging time)
 - 1.3.2.2. 110 s (60-s averaging time)
 - 1.3.2.3. 320 s (300-s averaging time)
 - 1.3.3. The measurement range of the Model 450I is from 0 to 10,000 ppb (10 ppm).
- 1.4. The stated precision of the 450I is 1% of reading or 1 ppb (whichever is greater).

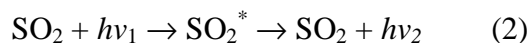
2. Summary of Method

Hydrogen sulfide in sample air entering the 450I is converted to sulfur dioxide (SO₂) in a converter operating at 300-400°C (1).



The sample flows directly to the hydrocarbon kicker when the 450I measures SO₂, or flows through the converter first, then to the hydrocarbon kicker when the 450I measures H₂S (Fig. 1). Any hydrocarbon present in the sample permeates through the tube wall of the hydrocarbon kicker, and is removed from the sample. SO₂ passes through the hydrocarbon kicker into the fluorescence chamber (the L-shaped box at the center right of Fig. 1).

The sample containing SO₂ is pulsed with ultraviolet (UV) light by a high-intensity xenon lamp. SO₂ molecules absorb at one wavelength and become excited (SO₂^{*}); they then decay to a lower energy state by emitting UV light at a different wavelength (2).



Emitted UV light is proportional to SO₂ concentration and is detected and converted to an electrical current by a PMT detector.

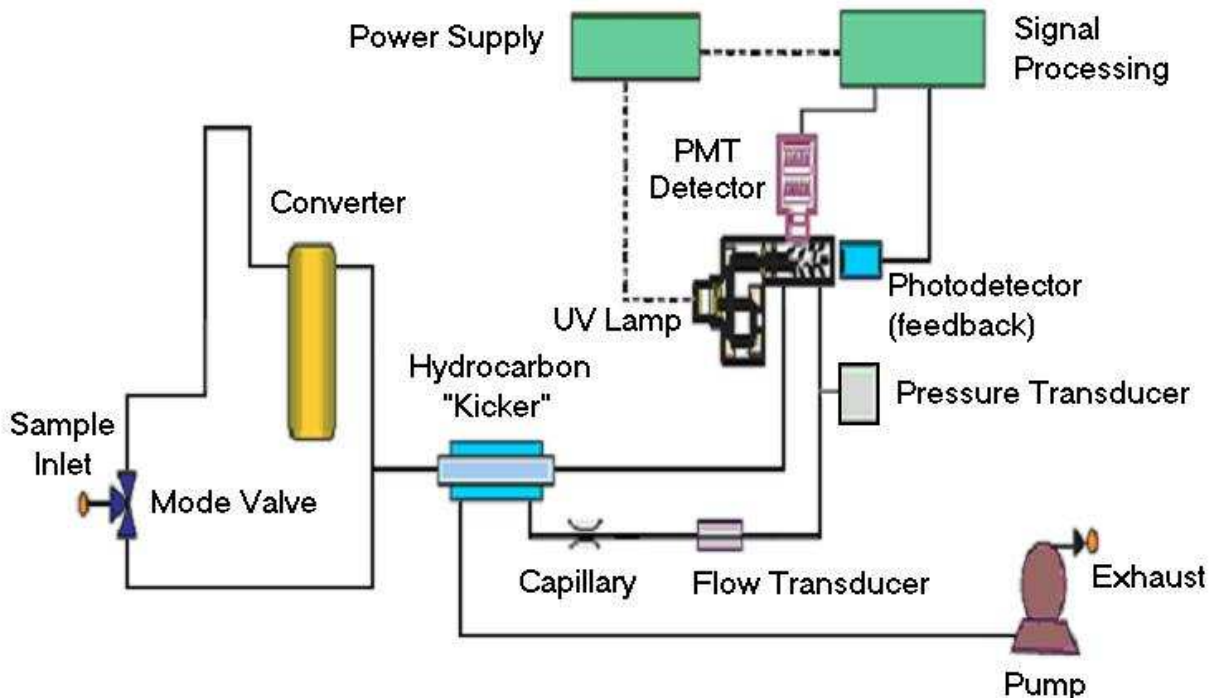


Figure 1. Flow schematic of the 450I.

3. Definitions of Terms and Acronyms

AirDAC	Data acquisition and control software for barn applications (SOP B2)
CS	Combined sulfur ($H_2S + SO_2$)
CAFO	Confined animal feeding operation
GSS	Gas sampling system (SOP G1)
Hydrocarbon kicker	A membrane tube in the 450I that removes aromatic hydrocarbons based on their ability to pass through the membrane. SO_2 molecules pass through the hydrocarbon kicker unaffected.
QAPP	Quality Assurance Project Plan
OFIS	On-farm instrument shelter (SOP U1)
PAAQL	Purdue Agricultural Air Quality Laboratory
PMT	Photomultiplier tube

4. Health and Safety

- 4.1. Be careful when working with the electrical power connection.
- 4.2. The converter of the Model 450I contains high-temperature elements ($300-400^\circ C$), and should not be touched.
- 4.3. Properly vent analyzer exhaust to avoid exposure to noxious gases in the sample.
- 4.4. Prevent leaking of the calibration gases into the working environment.

5. Cautions

- 5.1. Use antistatic wrist strap or discharge your body's electrostatic charge to a grounded metallic frame before touching any internal components of the 450I to avoid damage of the electronics by static discharge. See Chapter 7 ("Servicing") of the 450I manual.

6. Interferences

- 6.1. Compounds that interfere with fluorescence-based H₂S measurements are as follows:

Interferences	Effect on H ₂ S Readings
Methyl mercaptan	0.8 ppm increase per 1 ppm of CH ₃ SH
Dimethyl disulfide	0.5 ppm increase per 1 ppm of CH ₃ SSCH ₃
Dimethyl sulfide	< 0.1 ppm increase per 1 ppm of CH ₃ SCH ₃
NO	Increase of < 3 ppb at 500 ppb NO
<i>m</i> -xylene	Increase of < 1 ppb at 200 ppb <i>m</i> -xylene
Water	< 2% of reading per 2% humidity ratio

- 6.1.1. The effect of humidity will typically be less than 3% of reading. For example, the difference in humidity ratio between saturated air at 30°C (86°F) and saturated air at 0°C (32°F) is approximately 2.3%
- 6.1.2. H₂S is expected to be present at one or two orders of magnitude higher than dimethyl sulfide or dimethyl disulfide; thus, the relative magnitude of these interferences should be minor. Dimethyl sulfide and dimethyl disulfide can be monitored with the Innova Model 1412 Multi-Gas Monitor (SOP G7).
- 6.1.3. Concentrations of NO and *m*-xylene are expected to be negligible.

7. Personnel Qualifications

- 7.1. Personnel should be trained in the operation, maintenance and calibration of the analyzer before initiating the procedure. Training requires about three hours.
- 7.2. Each analyst must read and understand the entire analyzer manual and this SOP before operating the Model 450I.
- 7.3. Personnel should also be trained to interpret the analyzer output signal, converted concentration data, and correction of concentrations based on calibration.

8. Equipment and Supplies

- 8.1. Hydrogen sulfide analyzer (Model 450I, Thermo Electron Corporation)
- 8.2. H₂S-to-SO₂ converter (Model 340, Thermo Electron Corporation)
- 8.3. Keep the following spare parts at each site:
 - 8.3.1. 20-mil capillary, Part No. 4126
 - 8.3.2. O-ring capillary, Part No. 4800
 - 8.3.3. Teflon tubing (1/4" OD x 1/8" ID) for connecting analyzer to GSS and exhaust port
 - 8.3.4. 4-conductor cable (shielded, 22-gauge)

- 8.4. Keep the following spare parts in a central stock at PAAQL:
 - 8.4.1. 20-mil capillary, Part No. 4126
 - 8.4.2. O-ring capillary, Part No. 4800
 - 8.4.3. Pump, 110 VAC, Part No. 8550
 - 8.4.4. Pump repair kit, Part No. 8606
 - 8.4.5. Flash lamp, Part No. 8666
 - 8.4.6. Particulate filter assembly, Part No. 60100
 - 8.4.7. Converter assembly, 120 VAC, Part No. 450P702-1
 - 8.4.8. Power supply board, Part No. 10570
 - 8.4.9. Trigger pak, Part No. 8774
 - 8.4.10. Flash intensity board, Part No. 8884
 - 8.4.11. Flasher supply board, Part No. 9681
 - 8.4.12. Temperature control board, Part No. 8765
 - 8.4.13. PMT high voltage power supply, Part No. 9901
 - 8.4.14. Temperature control board (converter), Part No. 9889
 - 8.4.15. Teflon tubing (1/4" OD x 1/8" ID) for connecting analyzer to GSS and exhaust port
 - 8.4.16. 4-conductor cable (shielded, 22-gauge)
- 8.5. Calibration gas cylinders: Hydrogen sulfide (H₂S), sulfur dioxide (SO₂), zero gas (see the QAPP for exact specifications and compositions), with appropriate regulators (SOP G2)
 - 8.5.1. In projects where SO₂ is not a primary measurement, it may still be used to check the converter efficiency (Section 9.3).

9. Procedure

- 9.1. Model 450I Analyzer setup
 - 9.1.1. Check the exterior of the analyzer (Fig. 2) carefully to assure it is free of visible defects or damage.
 - 9.1.1.1. Remove the instrument cover to expose the internal components (Fig. 3).
 - 9.1.1.2. Remove any packing material, check for possible shipping damage and ensure that all connectors and circuit boards are firmly attached.
 - 9.1.1.3. Replace the instrument cover.
 - 9.1.2. Securely mount the analyzer in the instrument rack.
 - 9.1.3. Connect the analyzer manifold in the GSS to the "SAMPLE" bulkhead (Fig. 2) on the instrument's rear panel using 1/4" OD (1/8" ID) Teflon tubing less than 10' long. Allow enough slack to permit movement of the instrument rack.
 - 9.1.4. For barn applications:
 - 9.1.4.1. Connect the "EXHAUST" bulkhead to the OFIS air exhaust with 1/4" OD (1/8" ID) tubing less than 10' long, which is unimpeded and free of kinks or blockages.
 - 9.1.4.2. Connect a 4-conductor, 20- or 22-gage shielded cable to the designated channels of the FieldPoint analog input module (SOP B1). Properly ground the shield of the analog output cable.
 - 9.1.4.3. Connect the other end of the 4-conductor cable to the analyzer's analog outputs for SO₂ and H₂S (not to the CS analog output).
 - 9.1.4.3.1. Remove about 1.8 cm of insulation from the other end of the cable. Fold back the shielding. If necessary, use electrical tape or shrink tubing to hold

shielding in place. Expose a minimum of 1.5 cm of shielding. Strip each signal wire and connect them to the terminals (1) through (4) of the 8-position header of the 450I (Fig. 4) according to the wire connection to the data acquisition hardware

9.1.5. For open source applications:

9.1.5.1. Connect the “EXHAUST” bulkhead to the GSS bypass air exhaust with 1/4” OD (1/8” ID) tubing such that total tubing length is less than 10’ long. Tubing must be unimpeded and free of kinks or blockages.

9.1.5.2. Connect a null modem RS232 cable from the RS232 connector of the data-collecting computer to the “RS232/485” connector at the back of the instrument.



Figure 2. Front (top) and back (bottom) views of the TEC 450I.

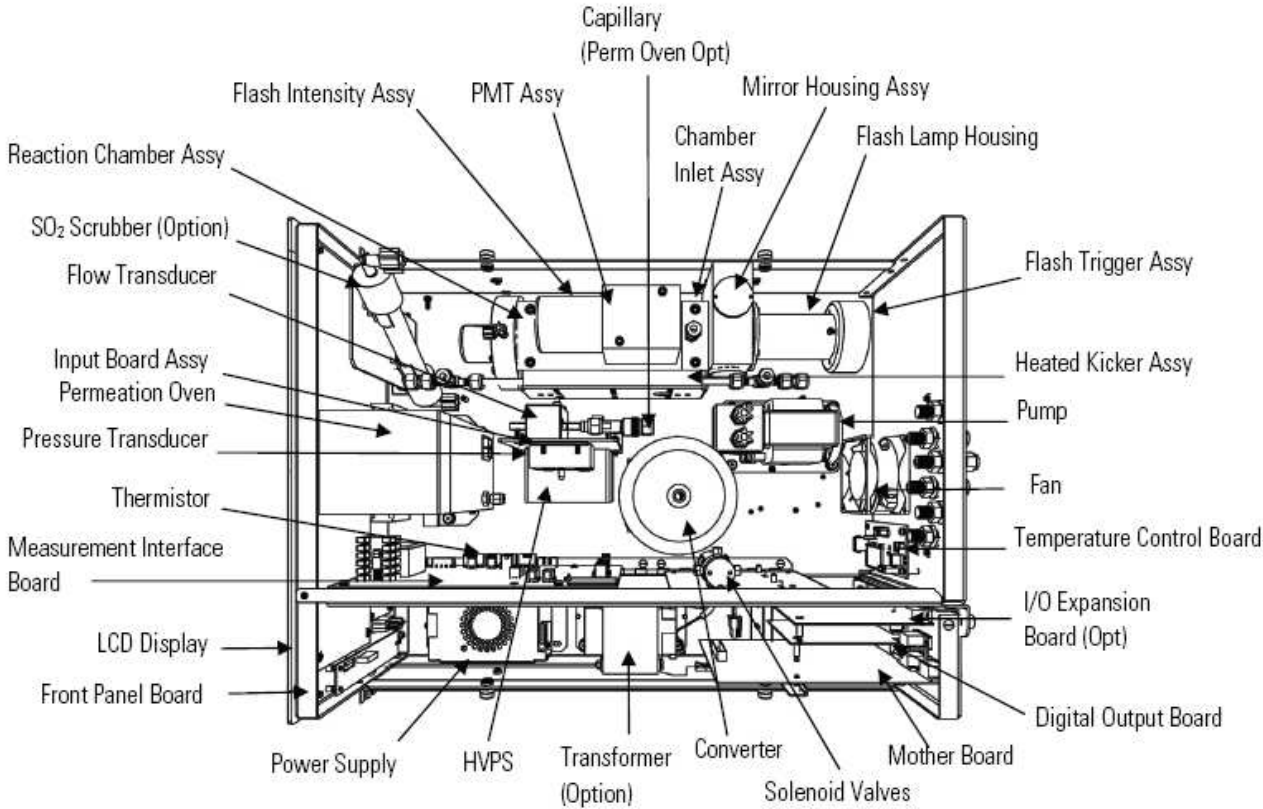


Figure 3. Internal schematic of the TEC 450I analyzer.

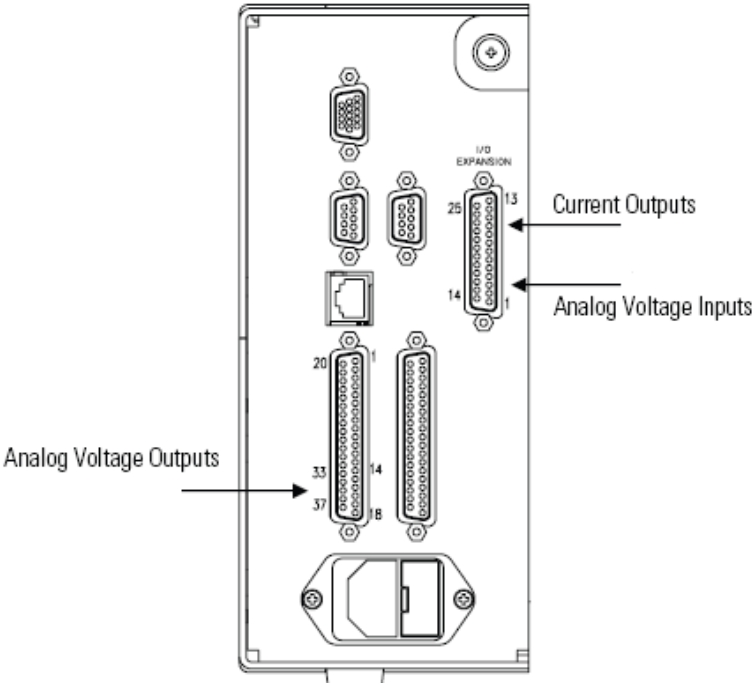


Figure 4. Connectors on the 450I rear panel (left side of Fig. 2 lower panel).

- 9.1.6. Plug the 450I power cord into a 110VAC power source, turn on its power, and allow a minimum of 90 min for the unit to warm up and stabilize before calibrating.
- 9.1.7. Operate the instrument menu (Fig. 5) to navigate to the screens necessary to set the instrument control settings (Table 1) and to check the operating parameters and ranges (Table 2), as described in detail in Chapter 3 of the instrument manual. An alarm message will appear in the screen on the front panel of the instrument when any of these parameters is operating outside the set limits.

Table 1. TEC 450I Instrument settings.

Parameter	Setting
Gas Units	ppb
Measurement Mode	Automatic Mode
Averaging Time	60 s
Temperature Correction	ON
Pressure Correction	ON
Flash Lamp	ON
Baud Rate	9600
Service Mode	OFF
Time	Current
Date	Current
Converter Temperature	352°F

Table 2. TEC 450I instrument operating parameters and ranges.

Parameter	Range
Pressure (mm Hg)	400 – 1000
Sample Flow (L/min)	0.350 – 1.400
Lamp Voltage (V)	500 – 1200
Lamp Frequency (Hz)	10,000 – 50,000
Converter Temperature (°C)	300 – 400
Internal Temperature (°C)	15 – 45
Chamber Temperature (°C)	43 – 47

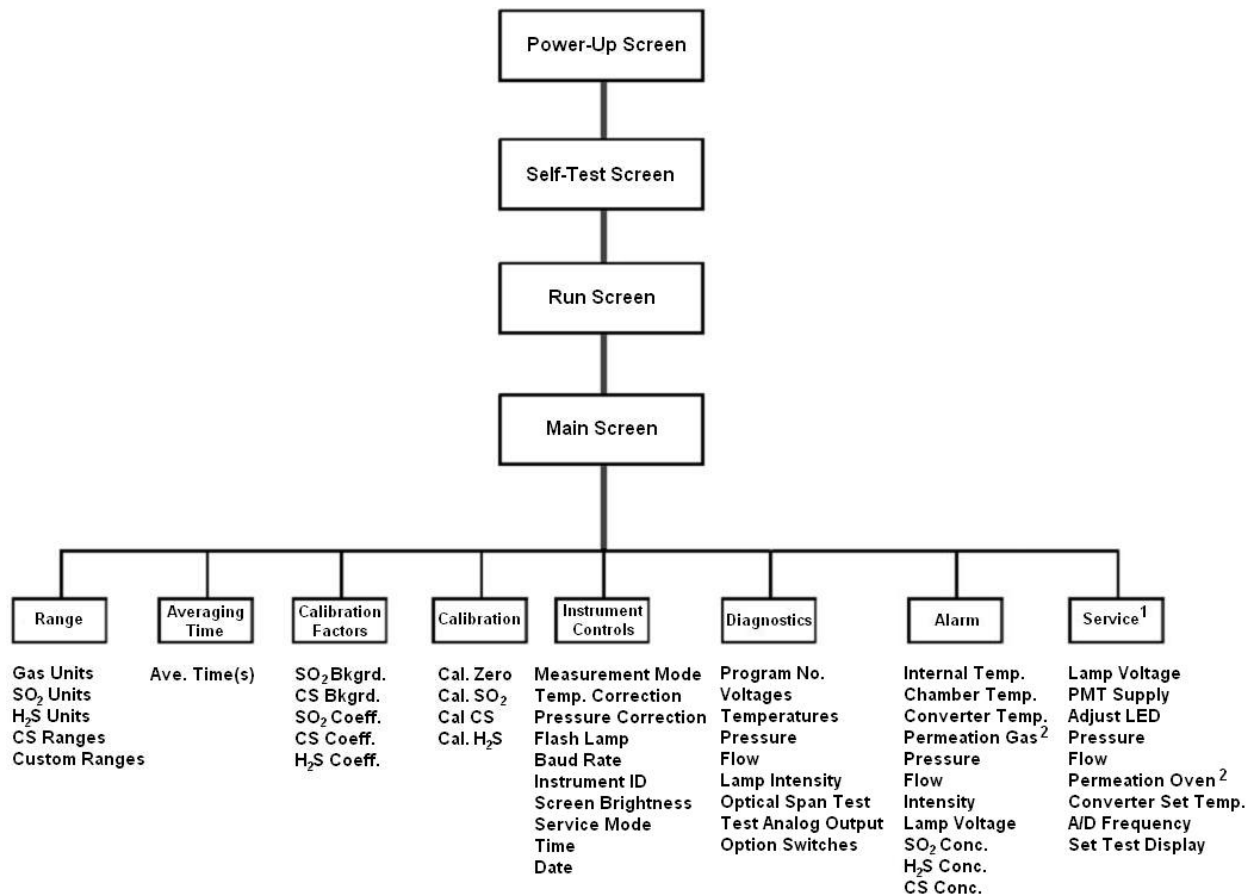


Figure 5. Partial flow chart of the TEC 450I menu from the instrument manual. Notes: 1 – Option only appears when the Service Mode is ON. 2 – Appears only when optional Permeation Oven is installed.

9.1.8. For barn measurements:

9.1.8.1. The analyte (H₂S, SO₂, CS) measurement ranges for the 450I are from 0 to 10,000 ppb. The analyte measurement range settings for each site are listed in the QAPP.

9.1.8.2. Enter the analyzer's analog signal ranges (0 to 10 VDC) and H₂S and SO₂ measurement ranges into the DAQ table in the AirDAC software (SOP B2) so that concentration reading can be displayed, converted and recorded.

9.1.9. For open source measurements:

9.1.9.1. Using iPort software, set data logging settings in instrument

9.1.9.1.1. Under "Main Menu->Instrument Controls->Datalogging settings":

9.1.9.1.1.1. Set "Select SREC/LREC" to "LREC"

- 9.1.9.1.1.2. Under “Select Content”, make sure the following appears
- | | | |
|-----------|---------|-------------------------------|
| “FIELD 1 | H2S” | |
| “FIELD 2 | SO2” | |
| “FIELD 3 | INTT” | (corresponds to INT TEMP) |
| “FIELD 4 | RCTT” | (corresponds to CHAMBER TEMP) |
| “FIELD 5 | CONVT” | (corresponds to H2S CNV TEMP) |
| “FIELD 6 | PRES” | (corresponds to CHAMBER PRES) |
| “FIELD 7 | SMPLFL” | (corresponds to SAMPLE FLOW) |
| “FIELD 8 | PMTV” | (corresponds to PMT VOLTS) |
| “FIELD 9 | LMPV” | (corresponds to FLASH VOLTS) |
| “FIELD 10 | LMPI” | (corresponds to FLASH REF) |
| “FIELD 11 | NONE” | |
| | | |
| “FIELD 32 | NONE” | |
- 9.1.9.1.1.3. Select any fields that are incorrect and modify as necessary. The item type for fields 1 and 2 is “CONCENTRATIONS” while that for the remaining 8 fields is “OTHER MEASUREMENTS”
- 9.1.9.1.1.4. *If changes were made to any of the above fields and if all data on the instrument have been saved to the LAN:* Go to “Main Menu->Instrument Controls->Datalogging Settings->Commit Content”; You will be asked to confirm that you want to commit content. Only say “YES” if you are positive that all data logged on the analyzer have been saved
- 9.1.9.1.1.5. Check configuration of the data logging at “Main Menu->Instrument Controls->Datalogging Settings->Configure Data logging”
- LOGGING PERIOD, MIN should be set to 1 MIN
 - MEMORY ALLOCATION SHOULD BE SET TO 100%
 - DATA TREATMENT should be set to AVG
- 9.1.9.1.1.6. Check the time zone setting; “Main Menu->Instrument Controls->Timezone”; should be set to UTC(GMT).
- 9.1.9.1.1.7. Set Ethernet communications at “Main Menu->Instrument Controls->Communication Settings->TCP/IP Settings->”
- >USE DHCP: OFF
 - >IP ADDR: 192.168.0.15
 - >NETMASK: 255.255.255.0
 - >GATEWAY: 192.168.0.1
 - >NTP Server: 132.239.1.6
- 9.1.9.1.1.8. If any value is modified, turn the instrument off and back on again.
- 9.1.9.2. Using the iPort software, prepare instrument for transmission and storage of the analyzer’s quality assurance and gas concentration measurements
- 9.1.9.2.1. Under “File->Preferences>instrument”:
- 9.1.9.2.1.1. Set “size” to FULL
- 9.1.9.2.1.2. Set “Data Record Loading” to “Save to File” and “Create”
- 9.1.9.2.2. Under “Comm->Connect”, assure settings are:
- Location: H2Sanalyzer
 - Com: (set to correct value)

Baud Rate: 115200
 Len: 8
 Stop: 1
 Parity: N
 Handshake: None
 Polling Config: 45 (this is the instrument ID)

- 9.1.9.2.3. Click on “OK” and respond “YES” to attach instrument.
- 9.1.9.2.4. Real-time data file save is set up under “Instrument->Load Records”: Click on “Real time” and click “Save to file”. Remove dash and spaces from the default file name (before the date and before the time components) and add the path and site code before the filename (e.g. C:\NAEMS\Data\OK4A\Period3\H2S\OK4A), and click “OK”
- 9.1.9.3. Verify that the output file is being produced in the desired location and check the contents by right clicking and opening with ‘Windows Notepad’. File should appear as:

```
;;
;; C:\NAEMS\Data\450I0103082156.dat Thu Jan 03 21:57:05 2008
;;
;; Model 450I- (prog iSeries 450I 01.05.06.165)
;;
Time Date Flags h2s so2 intt rctt convt pres smplfl pmtv lmpv lmpi
14:53 01-03-08 LL----- 1.052 9.36528 10.8132 32.8277 322.29 661.186 1.06992 -647.131 808.951 89
14:54 01-03-08 LL----- 1.48701 8.50626 10.9028 33.1631 324.389 656.023 1.06526 -647.131 809.76 88
14:55 01-03-08 LL----- 1.40748 7.61518 11.0148 33.4469 327.276 663.312 1.07233 -647.131 809.356 87
14:56 01-03-08 LL----- 0.247904 7.33171 11.1044 33.8854 326.751 658.149 1.06647 -647.131 810.973 88
14:57 01-03-08 LL----- 0.142362 7.49943 11.2164 34.066 323.865 663.312 1.07233 -647.131 808.143 88
14:58 01-03-08 LL----- 0.912892 7.91842 11.2612 34.1692 322.552 656.023 1.06492 -646.761 809.356 89
14:59 01-03-08 LL----- 0.957739 8.52361 11.3956 34.4788 325.177 661.186 1.07026 -647.131 808.547 89
15:02 01-03-08 LL---LL- 0 0 11.642 35.132 323.602 661.489 1.07164 -647.131 808.547 88
15:03 01-03-08 LL----- 0 0 11.754 35.352 323.077 655.719 1.06544 -646.761 807.738 88
```

9.2. Gas Analyzer Calibration

9.2.1. Reference calibration gases

9.2.1.1. Sulfur Dioxide (SO₂) in N₂ - exact composition as specified in the QAPP.

9.2.1.2. Hydrogen Sulfide (H₂S) in N₂ or air - exact composition as specified in the QAPP.

9.2.1.3. Zero air, also referred to as Continuous Emission Monitoring (CEM) Zero Air.

9.2.2. For barn measurements, set AirDAC to “Gas Cal” (Gas Calibration) and calibrate the 450I following SOPs G8 and G9

9.2.3. For open source measurements:

9.2.3.1. Start the 450I gas analyzer and let it warm up for at least 30 minutes.

9.2.3.2. Use Environics Series 4040 Gas Dilution System or TEC 146I dilutor to dilute H₂S levels if necessary.

9.2.3.2.1. Install gas line from exhaust of 450I to vent into trailer or outside

9.2.3.2.2. Install gas line from output port of dilutor to sample port of 450I, with a tee in the line to vent excess to outside through a flowmeter; this prevents over-pressurizing the 450I and verifies sufficient flow for 450I sampling.

9.2.3.2.3. Open both gas cylinders (calibration gas and zero air) to a pressure of 20 psi, then

9.2.3.2.4. Initial calibration gas is 0 ppm H₂S.

- 9.2.3.2.5. If using the Environics dilutor:
- 9.2.3.2.5.1. Attach gas lines of H₂S cylinder and Zero Air cylinder to ports 2 and 1 (respectively) of Environics dilutor.
 - 9.2.3.2.5.2. Set the H₂S concentration in the Environics dilutor program to the chosen concentration according to SOP G11.
- 9.2.3.2.6. If using the TEC 146I dilutor:
- 9.2.3.2.6.1. Attach gas lines from calibration cylinder to port A (or other programmed Port), and from the zero air cylinder to Zero Air Port.
 - 9.2.3.2.6.1.1. Connect gas line from zero air cylinder (for 0 ppm H₂S) to sample port of 450I, with a tee in the line that vents exhaust (this prevents over-pressurizing the 450I).
 - 9.2.3.2.6.2. Set the H₂S concentration on the dilutor (not 0 ppm) to the chosen concentration according to SOP C3.
- 9.2.3.2.7. Open flow valve of regulator. Flow calibration gas through the 450I and wait 5-10 min. for the H₂S concentration reading on the 450I to stabilize.
- 9.2.3.2.8. Repeat sections 9.2.3.2.4, 9.2.3.2.6 (Environics dilutor) or 9.2.3.2.5, 9.2.3.2.6 (TEC 146i dilutor) using calibration gas at appropriate concentration.
- 9.2.3.2.8.1. Record pressure, flow rate, lamp intensity, and lamp voltage from 450I on a form similar to Table 3.
 - 9.2.3.2.8.2. Once stabilized record H₂S output concentration from dilutor and H₂S concentration reading on the 450I.
- 9.2.3.3. If multipoint calibration, repeat section 9.2.3.2 for each target concentration, running a zero test in between span checks (the four concentrations are 3ppm, 2ppm, 1ppm, and 0.5ppm) in decreasing concentration and record results on form similar to Table 3.
- 9.2.3.3.1. Calculate linear regression of instrument response to concentration and apply to records as a post-process.
- 9.2.3.4. If calibration verification check, repeat section 9.2.3.2 for zero and 0.5 ppm concentrations respectively.

Table 3 – Example Multi-point calibration form for Open source instruments

450I Gas Analyzer Multi-Point Calibration								
Serial #	Time	Pressure	Flow	Lamp Int.	Lamp Volt.	H ₂ S cyl. Conc.	Dilutor Conc.	450I H ₂ S reading
						0		
						3.0		
						0		
						2.0		
						0		
						1.0		
						0		
						0.5		
						0		

- 9.2.4. Recording information during a calibration or precision check
- 9.2.4.1. Record the analyzer's responses to both zero and reference (precision check) gases before and after adjusting the analyzer. The recorded responses before adjustment (as-found responses), are used to correct the data since the previous calibration or previous precision checks.
- 9.2.4.2. Record 450I pressure, sample flow, lamp intensity, and lamp voltage in the electronic field notes in a table like Table 4.
- 9.2.4.3. For barn measurements: Record the start / stop (read) times, analyzer, calibration gas, calibration gas concentration, analyzer reading, AirDAC concentration reading, and AirDAC signal in the field notes like in Table 5 (Precision Checks) or Table 6 (Calibration)
- 9.2.4.4. For open source measurements: Record the start / stop (read) times, analyzer, calibration gas, calibration gas concentration, analyzer reading, data file concentration reading in the field notes like in Table 7 (Calibration Verification/Precision Checks).
- 9.2.5. Introduce reference gas. The number of points and gas concentrations for multipoint calibrations and calibration verification/ precision checks are specified in the Quality Objectives of the QAPP.
- 9.2.5.1. Start flow of reference gas to the analyzer. See SOP G1 for delivery options
- 9.2.5.2. Record date and time in the electronic field notes in a table like Table 5.
- 9.2.5.3. Record pertinent gas delivery parameters (SOP G1).
- 9.2.5.4. Wait a minimum of 10 min for the display to stabilize (The minimum of 10 min corresponds with the sampling time used for each location).
- 9.2.5.5. Record the time and concentrations shown on the 450I display, and in AirDAC (barn measurements), on provided form and Excel® spreadsheet form (open area measurements) and in the electronic or paper field notes. Update the control chart in accordance with SOP Q1.

Table 4. Recording of TEC 450I operating parameters.

Date	TEC 450I Operating Parameters			
	Pressure,	Sample Flow	Lamp Intensity	Lamp Voltage
	mm Hg	L/min	Hz	V
3/07/2006	558.6	0.996	21173	1095
3/12/2006	557.5	1.040	20998	1094
“	“	“	“	“
“	“	“	“	“

- 9.2.5.6. If the stabilized concentration during a precision check deviates from the control chart baseline calibration value by 10% or more, conduct a multipoint calibration in accordance with SOP G9 (barn measurements) or Section 9.2.3.3 of this SOP (open area measurements).
- 9.2.5.7. Stop gas flow and record the time.
- 9.2.6. Repeat the steps in Section 9.2.5. for each reference gas.
- 9.2.7. Return analyzer's display back to the run screen by pressing run.

- 9.2.8. For barn measurements, return the GSS back to normal sampling mode by setting AirDAC to auto sample.
- 9.3. Converter Efficiency Check
 - 9.3.1. Following Section 9.2, calibrate the SO₂ and CS channels with the SO₂ calibration gas. Then, calibrate the H₂S channel with the H₂S calibration gas.
 - 9.3.2. Navigate to the “Calibration Factors” Menu (Fig. 5). The H₂S coefficient shown here is the converter efficiency.
 - 9.3.2.1. For example, if the H₂S coefficient is 0.905, the converter efficiency is 90.5%.
- 9.4. Maintenance
 - 9.4.1. Preventive maintenance is covered in Chapter 5 of the 450I Instrument Manual.
 - 9.4.1.1. Clean the dust filters on the back of the analyzer and converter on a regular basis. This can range from weekly to monthly, depending on the dust level in the OFIS.
 - 9.4.1.2. If the lamp voltage is equal to or greater than 1200 V, lower the voltage by adjusting the potentiometer (R28) on the lamp power supply board (See Chapter 7 of the Instrument Manual for Servicing), or replace the lamp.
 - 9.4.1.3. Run a leak test if the sample flow rate, as reported by AirDAC (barn measurements) or iPORT (open area measurements), is < 0.9 L/min.
 - 9.4.1.3.1. Block the bulkhead labeled “SAMPLE” on rear panel with a leak-tight cap then wait 5 to 10 min.
 - 9.4.1.3.2. Check the sample flow and pressure readings. There should be zero flow and less than 160 mm Hg pressure. If not, check to see that none of the fittings or threads of the input lines are cracked or broken.
 - 9.4.1.4. Check the capillary tubes for particulate deposits within the bore. Clean or replace as necessary.
 - 9.4.1.5. Conduct a zero & precision span check before and after any maintenance, except for cleaning of the dust filter.
- 9.5. Troubleshooting
 - 9.5.1. Troubleshooting guidelines are in Chapter 6 of the 450I manual.
 - 9.5.1.1. Check wiring, power connections, and fuses if there is no signal or power.
 - 9.5.1.2. Check flow path integrity if span concentration cannot be attained, or if flow rate is low.
 - 9.5.1.3. If there is no or low flow, check the capillary tube or internal pump.
 - 9.5.1.4. Replace lamp if the lamp intensity is varying widely (ranging quickly from 10,000 to 50,000 Hz).
- 9.6. Data acquisition, calculations & data reduction requirements
 - 9.6.1. Barn measurements: Monitor a 0-10 VDC analog output signal using AirDAC (SOP B2).
 - 9.6.2. Open area measurements: Monitor measurements using iPort software.
 - 9.6.3. Report all data with 3 significant digits.
 - 9.6.4. Follow SOP B4 to correct gas concentrations for calibration and air density.
- 9.7. Computer hardware & software
 - 9.7.1. Barn measurements:
 - 9.7.1.1. DAQ hardware (SOP B1)
 - 9.7.1.2. AirDAC (SOP B2)
 - 9.7.1.3. ARDIS (SOP B3)

- 9.7.1.4. CAPECAB (SOP B6)
- 9.7.2. Open area measurements:
 - 9.7.2.1. LAN server (SOP U4)
 - 9.7.2.2. iPort software

10. Data and Records Management

- 10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
- 10.2. Manage all data according to SOP B5 (barn measurements) or SOP D1 (open area measurements).
- 10.3. Document all data and information (e.g., sample collection method used) on field data sheets, and within site logbooks with permanent ink, or in electronic field notes.
- 10.4. Overstrike all errors in writing with a single line, and initial and date all such corrections.
 - 10.4.1. Open area measurements:
 - 10.4.1.1. LAN server (SOP U4)
 - 10.4.1.2. iPort software

11. Quality Control and Quality Assurance

- 11.1. Conduct zero and one or more precision checks at the QAPP-specified frequency.
- 11.2. Conduct multipoint calibrations at the QAPP-specified frequency
- 11.3. Conduct a multipoint calibration whenever a precision check indicates a drift of $> \pm 10\%$ from the baseline calibration value, unless otherwise stated in the QAPP for a particular project.
- 11.4. Conduct a multipoint calibration whenever the zero check is $> \pm 5\%$ of full scale different than the baseline zero, unless otherwise stated in the QAPP for the particular project.
- 11.5. Check response time every month for barn measurements, and every six months for open area measurements.
- 11.6. In cases where SO_2 is not a primary measurement, weekly zero/precision checks with SO_2 are not necessary. In these cases, the QAPP will dictate the frequency of converter efficiency checks, and the converter efficiency that must be maintained.
- 11.7. Perform preventative maintenance every six months, according to the schedule provided in Chapter 5 of the 450I manual.
 - 11.7.1. Visually inspect for obvious visible defects (loose connections and/or fittings, cracked or clogged Teflon lines, and excessive dust accumulation).
 - 11.7.2. Inspect the fan filter located on the back of the instrument weekly; clean as needed.
 - 11.7.3. The schedule for replacing the sample particulate filter is found in the QAPP.
 - 11.7.4. Measure the gas analyzer inlet flow rate every six months.
 - 11.7.5. Check the lamp voltage under the diagnostics menu. If the lamp voltage is 1200 V or higher, replace the lamp or adjust its voltage.
 - 11.7.6. Leak check the sample line and flow components and fittings every six months.

- 11.8. If troubleshooting of the analyzer reveals particulate deposits in the capillaries, these must be replaced.

12. References

- 12.1. iPort Instruction Manual. 2007. *iPort Instruction Manual*. Communications software for iSeries and C Series Instruments. Part#102606-00. Thermo Fisher Scientific, Franklin, MA.
- 12.2. TFS 450I Manual. 2006. Model 450I Pulsed Fluorescence SO₂- H₂S-CS Analyzer Instruction Manual. Part #103258-00. Thermo Fisher Scientific, Franklin, MA.
- 12.3. SOP B1. 2007. Data Acquisition and Control Hardware. Standard Operating Procedure B1, v. 1.0. Purdue Ag Air Quality Lab.
- 12.4. SOP B2. 2007. Data Acquisition and Control Software (AirDAC). Standard Operating Procedure B, v. 1.02. Purdue Ag Air Quality Lab.
- 12.5. SOP B3. 2006. Air Data Pre-Processing Software. Standard Operating Procedure B3, v. 0.0. Purdue Ag Air Quality Lab.
- 12.6. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5, v. 0.0. Purdue Ag Air Quality Lab.
- 12.7. SOP B6. 2007. Data Processing Software (CAPECAB). Standard Operating Procedure B6, v. 1.0. Purdue Ag Air Quality Lab.
- 12.8. SOP G1. 2006. The PAAQL Gas Sampling System. Standard Operating Procedure G1, v. 0.0. Purdue Ag Air Quality Lab.
- 12.9. SOP G2. 2006. Compressed Gas Cylinders. Standard Operating Procedure G2, v. 0.0. Purdue Ag Air Quality Lab.
- 12.10. SOP G7. 2008. Use of the INNOVA 1412 Photoacoustic Multi-Gas Monitor. Standard Operating Procedure G7, v. 1.0. Purdue Ag Air Quality Lab.
- 12.11. SOP G8. 2007. Multi-Point Calibration of Gas Analyzers. Standard Operating Procedure G8, v. 1.0. Purdue Ag Air Quality Lab.
- 12.12. SOP G9. 2006. Zero and Precision Checks of Gas Analyzers. Standard Operating Procedure G9, v. 0.0. Purdue Ag Air Quality Lab.
- 12.13. SOP G11. 2007. Operation of the Environics[®] Computerized Gas Dilution System. Standard Operating Procedure G11, v. 1.0. Purdue Ag Air Quality Lab.
- 12.14. SOP Q1. 2007. Use of Control Charts for Performance Monitoring of Gas Analyzers and Analytical Instruments. Standard Operating Procedure Q1, v. 1.0. Purdue Ag Air Quality Lab.

13. Contact Information

- 13.1. Manufacturer: Thermo Fisher Scientific (www.thermo.com), 8 West Forge Parkway, Franklin, MA 02038, Technical Support: 1-866-282-0430
- 13.2. PAAQL: odor@purdue.edu, heber@purdue.edu, <http://www.AgAirQuality.com>.

Table 5. TEC 450I Analyzer Precision Check - Barn Measurements.

Precision Checks							
3/12/2006				Concentration	Instrument	Lab View	Signal
Started	Stopped	Instrument	Gas	ppb	Reading	ppm /ppb	vdc or mA
4/28/2006 14:05	4/28/2006 14:15	TEC 450I (H2S)	CEM Zero	0	-3.1	2.55	0.0005
4/28/2006 14:05	4/28/2006 14:15	TEC 450I (SO2)	CEM Zero	0	0	0.98	0.0008
4/28/2006 14:16	4/28/2006 14:26	TEC 450I (H2S)	H2S	2080	2090	2101.22	2.090
4/28/2006 14:16	4/28/2006 14:26	TEC 450I (SO2)	H2S	2080	10	10.78	0.011
4/28/2006 14:27	4/28/2006 14:37	TEC 450I (H2S)	SO2	1900	-106	-107.74	-0.109
4/28/2006 14:27	4/28/2006 14:37	TEC 450I (SO2)	SO2	1900	1907	1904.71	1.912

Table 6. TEC 450I Analyzer Calibration Check- Barn Measurements.

Calibration (H2S)							
8/9/2005				Concentration	Instrument	Lab View	Signal
Started	Stopped	Instrument / Comments	Gas	ppb	Reading	ppb	vdc
8/9/2005 8:55	8/9/2005 9:10	TEC 450I	CEM Zero	0	-33	-38	0.059
8/9/2005 9:11	8/9/2005 9:22		H2S	4370	4570	4551	9.091
8/9/2005 9:23	8/9/2005 9:35	Recorded reading	CEM Zero	0	-25.7	-33	0.049
	8/9/2005 9:37	Zero-ed instrument		0			
	8/9/2005 9:39	Recorded reading (9:39:19)		0	0	0	0.001
8/9/2005 9:11	8/9/2005 9:22	Recorded reading	H2S	4370	4540	4536	9.071
	8/9/2005 9:52	Adjusted instrument to H2S span of 4370 ppb					
	8/9/2005 9:53	Recorded reading (9:53:08)			4370	4371	8.741
8/9/2005 9:54	8/9/2005 10:06		CEM Zero	0	3.6	5.0	0.011
8/9/2005 10:06	8/9/2005 10:17		H2S	4370	4370	4376	8.751
8/9/2005 10:17	8/9/2005 10:28		CEM Zero	0	0.4	0	0.001
8/9/2005 10:28	8/9/2005 10:39		H2S	4370	4420	4416	8.831
8/9/2005 11:19	8/9/2005 11:29	Baseline	CEM Zero	0	-1.6	-2	0.001
8/9/2005 11:29	8/9/2005 11:39	Baseline	H2S	4370	4380	4361	8.771

Table 7. TEC 450I Analyzer Calibration Verification Check- Open Area Measurements.

3/12/2006			Concentration	Instrument Reading	Deviation from baseline	Site	FOS
Started	Stopped	Gas	ppb				
4/28/2006 14:05	4/28/2006 14:15	CEM Zero	0	-3.1			
4/28/2006 14:05	4/28/2006 14:15	CEM Zero	0	0			
4/28/2006 14:16	4/28/2006 14:26	H2S	2080	2090			
4/28/2006 14:16	4/28/2006 14:26	H2S	2080	10			

**MEASUREMENT OF METHANE AND NON-METHANE
HYDROCARBONS WITH THE THERMO ELECTRON
MODEL 55C ANALYZER**

Standard Operating Procedure (SOP) G6

**MEASUREMENT OF METHANE AND NON-METHANE HYDROCARBONS
WITH THE THERMO ELECTRON MODEL 55C ANALYZER
Standard Operating Procedure (SOP) G6**

**Prepared by
Bill W. Bogan and Claude A. Diehl**

**Reviewed by
Albert J. Heber**

Effective Date: November 6, 2007

**PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)
Agricultural and Biological Engineering, Purdue University
West Lafayette, IN 47907**

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions of Terms and Acronyms	4
4. Health and Safety	4
5. Interferences	4
6. Personnel Qualifications.....	5
7. Equipment and Supplies	5
8. Procedure.....	6
9. Data and Records Management	12
10. Quality Control and Quality Assurance	12
11. References.....	13
12. Contact Information	14

1. Scope and Applicability

- 1.1. The purpose of this procedure is to present the methodology and instrumentation used to measure automatically and sequentially concentrations of methane and non-methane hydrocarbons (NMHC) at animal feeding operations (AFO), specifically at barns.
- 1.2. This procedure applies only to the Thermo Electron Corporation (TEC) Model 55C Methane and Non-Methane Hydrocarbons (NMHC) Analyzer.
- 1.3. The TEC Model 55C NMHC analyzer uses automated gas chromatographic (GC) method that is useful for measuring both methane and NMHC at low concentrations in a short time and a wide measurement range.
- 1.4. The detection limit of the method is 20 ppb for methane and 50 ppb for NMHC (as propane).
- 1.5. The range is from 0 to 2000 ppm
- 1.6. The analysis time is 70 s.

2. Summary of Method

The Thermo Electron Model 55C is a back-flush gas chromatography (GC) system which provides automated measurement of both methane and non-methane hydrocarbons. The back-flush GC method employed by the Model 55C is unique in that it provides a direct, accurate measurement of non-methane concentrations, even when these are present at low levels in the presence of methane at much higher concentrations. The Model 55C is an automated batch analyzer which repeatedly collects and analyzes small amounts of the sample stream drawn in by the pump.

The Model 55C's measurement of methane and non-methane hydrocarbons utilizes a proprietary gas chromatography column, developed specifically for this application, which is capable of achieving complete separation of methane from all other hydrocarbons, including C₂ compounds. Due to its low molecular weight and high volatility, methane moves faster than other organic compounds and is the first to emerge from the opposite end of the column. Upon leaving the column, the methane flows to the flame ionization detector, or FID, which uses a hydrogen flame to ionize organic molecules in the carrier gas. The methane is measured by the FID and its signal is converted into a concentration by comparison with the signal produced by a calibration gas. Once the methane peak has been detected, the flow of carrier gas through the column is reversed, and non-methane hydrocarbons remaining on the column are "back-flushed" out and carried to the FID for measurement.

As the NMHCs reach the FID, they create a signal that is proportional to the total NMHC concentration and can be converted to a ppm reading by comparison with the signal generated by a known standard. If the FID signal is continuously monitored with a chart recorder, the output will create a chromatogram showing one peak for methane (which reaches a maximum approximately 17 s after injection), and a second peak that represents the combined NMHC. The NMHC peak will generally be lower and wider than the methane peak, and its retention times can vary somewhat depending on operating conditions and sample composition. The total time required for analysis of one sample is about 70 s.

3. Definitions of Terms and Acronyms

- | | | |
|------|-------|--|
| 3.1. | NMHC | Non-methane hydrocarbons |
| 3.2. | DAC | Data acquisition and control |
| 3.3. | FID | Flame ionization detector |
| 3.4. | GSS | Gas sampling system |
| 3.5. | PAAQL | Purdue Agricultural Air Quality Laboratory |
| 3.6. | TEC | Thermo Electron Corporation |

4. Health and Safety

- 4.1. Be careful when working with the electrical power connection.
- 4.2. Unplug the instrument before servicing any internal component.
- 4.3. The instrument is supplied with a three-wire grounding cord. Under no circumstance should this grounding system be defeated or tampered with.
- 4.4. During operation, the sample intake, sample bypass, and FID exhaust bulkheads are extremely hot. Use appropriate caution when working with a hot instrument.
- 4.5. Properly vent analyzer exhaust (FID exhaust and sample bypass) to avoid exposure to noxious gases
- 4.6. Be careful when lifting the analyzer. The analyzer weighs 60 lb.

5. Cautions

- 5.1. Do not apply power to the Model 55C until all other installation procedures have been completed. An incorrect start-up sequence could damage the instrument.
- 5.2. Some internal components can be damaged by the discharge of static electricity. To avoid damaging internal components, wear an antistatic wrist strap that is properly connected to earth ground when servicing any internal components of the 55C. If an antistatic wrist strap is not available, be sure to discharge static electricity by touching a grounded metal object before touching any internal components. Note that when the 55C is unplugged, its chassis is not grounded.
- 5.3. Support gases, regulators, gas lines, gas scrubbers, and fittings must be carefully selected to provide the cleanest possible support gases to the instrument. Use of inappropriate gas supplies or hardware will have an adverse affect on performance (e.g. cause baseline instabilities that interfere with the detection of low hydrocarbon concentrations), and can contaminate the instrument with oils or other organic compounds.
 - 5.3.1. Use only regulators with stainless steel diaphragms (generally referred to as high purity regulators). Do not use general purpose, industrial, or welding regulators that usually have a rubber or polymeric diaphragm. Polymeric diaphragms tend to give off organic compounds.
 - 5.3.2. Use only pre-cleaned, GC-grade copper or stainless steel tubing, purchased from a GC supply house. Tubing can also be coated with non-reactive SilcoSteel coating. Gas lines made of plastic materials such as Teflon® can give off organic vapors and are sometimes permeable to permanent gases.

- 5.3.3. Use 1/4" OD tubing and compression fittings (Gyrolock). It is important to use compression fittings. Do not mix different types of fittings, as this will cause leaks and unreliable performance of the TEC 55C analyzer.
- 5.3.4. Use either high-grade support gases (Section 8.5), or use appropriate gas scrubbers. Trace oxygen, hydrocarbons, and water vapor cause baseline instability and poor analyzer performance. Gas scrubbers are used to remove traces of oxygen, hydrocarbons, and water vapor from the support gases.
- 5.4. Actuator air must be delivered to the instrument at 40 to 45 psi. Air pressures in excess of 45 psi can reduce the service life of the valve actuator; pressures lower than 40 psi may cause erratic performance.

6. Interferences

- 6.1. Any hydrocarbon contamination of gases can lead to unstable baselines in the output chromatograph. Carrier gas must be completely free of hydrocarbons.
- 6.2. Depending upon composition of samples, it may be necessary to incorporate a delay so that other, non-hydrocarbon sample constituents can be flushed off the column to avoid interference with analyses of subsequent samples.
- 6.3. During calibration, methane concentration in span gas should never be less than approximately 1.5 ppm, as interference from oxygen can lead to inaccurate results.
- 6.4. Fluctuations of sample inlet pressures may cause problems with repeatability and accuracy of hydrocarbon measurements, unless the GSS is operating properly, and the analyzer manifold is maintaining a constant pressure.

7. Personnel Qualifications

- 7.1. Personnel should be trained in the use of the instrument before initiating the procedure.
- 7.2. Each analyst must read and understand the entire instrument manual and this SOP before operating the analyzer.
- 7.3. Personnel should also be trained to interpret the analyzer output signal, converted concentration data, and correction of concentrations based on calibration.

8. Equipment and Supplies

- 8.1. Methane/Non-Methane Analyzer (Model 55C, Thermo Electron Corporation).
- 8.2. Keep a copy of the 55C Instrument Manuals at each field location.
- 8.3. Keep the following spare parts at each remote site:
 - 8.3.1. Fuse, Part No. 14938
 - 8.3.2. Fuse, transformer fuse, Part No. 14011
 - 8.3.3. O-rings, Part No. 11032
 - 8.3.4. O-rings, Part No. 11030
 - 8.3.5. O-rings, FID kit Part No. 14818
- 8.4. Keep the following spare parts in a central stock at PAAQL:
 - 8.4.1. Fuse, Part No. 14938

- 8.4.2. Fuse, transformer fuse Part No. 14011
- 8.4.3. O-rings, Part No. 11032
- 8.4.4. O-rings, Part No. 11030
- 8.4.5. O-rings, FID kit Part No. 14818
- 8.4.6. Ignitor glow plug, Part No. 10981
- 8.4.7. Ignitor assembly, Part No. 15018
- 8.5. Pre-cleaned, 1/4" OD stainless steel or copper GC-grade tubing
- 8.6. Compression fittings (e.g. Gyrolock)
- 8.7. The following grades of gases are specified:
 - 8.7.1. Combustion Air – Hydrocarbon-free air: Must be <0.1 ppm hydrocarbons
 - 8.7.2. Actuator Air – Zero air
 - 8.7.3. FID Fuel – Hydrogen: 99.999% or higher purity as a fuel
 - 8.7.4. Carrier Gas – Ultrahigh purity nitrogen: 99.9995% pure (CEM grade) or higher
 - 8.7.5. Calibration Gas – Should be a mixture of methane (not less than 2 ppm) and propane (not less than 1 ppm) with an analytical accuracy of $\pm 1\%$.
- 8.8. Appropriate regulators for each gas cylinder (SOP G2)

9. Procedure

- 9.1. Instrument Setup
 - 9.1.1. When unpacking the Model 55C, save all packing material. If it is ever necessary to ship the instrument for service, the original packing material should be re-used. It is especially important to cap all gas fittings and to wrap the instrument in plastic before shipment to avoid contamination of the gas lines with packing foam.
 - 9.1.2. Check the exterior of the analyzer carefully to assure it is free of visible defects or damage.
 - 9.1.2.1. Remove the caps or plugs that cover the gas fittings on the rear panel.
 - 9.1.2.2. Remove the instrument cover to expose the internal components
 - 9.1.2.3. Remove any packing material, check for possible shipping damage and ensure that all connectors and circuit boards are firmly attached
 - 9.1.2.4. Replace the instrument cover
- 9.2. Connect the air, fuel, and carrier gas lines from the regulators on their respective cylinders to the rear panel bulkheads using 1/4" OD line (Section 5.3.2) and the compression fittings supplied with the instrument.
 - 9.2.1. In some models there may be two air inlets. In this case, the air inlet in the upper right quarter of the panel supplies the FID and the air inlet in the lower left quarter supplies the rotary valve actuator.
- 9.3. Connect the sample line from the GSS analyzer manifold to the bulkhead fitting labeled "SAMPLE INLET" using the 1/4" compression fitting that is provided.
- 9.4. The sample should be presented to the Model 55C at a stable pressure near normal atmospheric levels. Connect the 55C to the analyzer manifold of the GSS (SOP G1) to accomplish this. The instrument's internal pump can work against a vacuum of about 10 in of water, and will draw sample through at least 100-ft of 1/4" OD sample line. The length of tubing between the analyzer manifold and the analyzer itself will be less than 20 ft.

- 9.5. Connect the FID Exhaust (located in the lower-right corner of the rear panel next to the sample inlet) to a suitable vent. Use as short a line as possible. The diameter should be as large as possible to prevent pressurizing the FID. Since the exhaust contains steam, configure the line to slope away from the instrument to drain condensation.
- 9.6. Connect the “BY-PASS” port to a suitable vent. The connection should be as short as possible and should open at near-atmospheric pressure.
- 9.6.1. Do not connect the sample by-pass and the FID exhaust port to the same line.
- 9.6.2. Check the label on the rear panel to confirm that the instrument is properly configured for the local power supply, and use the supplied cord to connect the Model 55C to the appropriate outlet.
- 9.7. Instrument start-up
- 9.7.1. Turn on all gas cylinders and adjust cylinder regulators to the following pressures:

Fuel (hydrogen)	60 psi
Support air	45 psi
Carrier gas	60 psi
Span gas (calibration gas)	15 psi
Air drive valve actuator	45 psi

- 9.7.2. Slightly loosen each fitting at the rear of the instrument for about 30 s to bleed off air trapped in the lines and scrubbers, and the retighten the fittings.
- 9.7.3. Turn on the main power switch and note whether any error messages are reported. After a brief diagnostic test, the 55C will enter the “warm-up” mode. The instrument will require about 90 min to reach its full operating temperature. In order to avoid contamination of the sampling system, the pump automatically shuts off during part of the warm-up period.
- 9.7.4. While the instrument is warming up, check and adjust the carrier gas and air pressures using the digital display and pressure regulator controls located on the left side of the Model 55C front panel. The correct parameters are as follows:

Air	24.5 psi
Carrier	33.0 psi
Fuel	33.5 psi
Fuel type	H ₂
Detector oven	175 °C
Column oven	70 °C

- 9.7.4.1. Note that the fuel pressure will read “0” during warm-up because the fuel solenoid is automatically closed.
- 9.7.5. When the instrument has stabilized at the correct operating temperature, the fuel solenoid will automatically open and the Model 55C will attempt to light the FID.
- 9.7.5.1. If the ignition fails, the fuel will be shut off, and the front screen will prompt the user to press “ENTER” to try again.

- 9.7.5.2. During the ignition cycle, check the fuel pressure, and if necessary, adjust to match the value shown in the table above.
- 9.7.5.3. If the FID fails to light after repeated attempts, see the troubleshooting instructions in Chapter 5 of the instrument manual.
- 9.7.6. Turn the pump on using the switch on the front panel, and then press the RUN button to start automatic analyses using the factory entered parameters and calibration.
- 9.8. Column Conditioning and Initial Burn-in
 - 9.8.1. Run the built-in column conditioning routine after the start-up procedure. The column conditioning procedure is as follows:
 - 9.8.1.1. Press the "MENU" key to interrupt normal operation.
 - 9.8.1.2. After the Main Menu appears, use the "DOWN ARROW" key to move the cursor to item No.7, "SERVICE". Press "ENTER".
 - 9.8.1.3. Move the cursor down to item No. 4, "CONDITION COLUMN", and press "ENTER".
 - 9.8.1.4. The instrument will raise the oven temperature for 8 h to condition the column and clean out any accumulated hydrocarbons. After 8 h, the instrument will return to normal operating temperatures.
- 9.9. Running test chromatograms
 - 9.9.1. Following installation and burn-in, it is advisable to obtain a series of test chromatograms to ensure that the Model 55C is operating correctly. Generate test chromatograms at the installation of a new column or new instrument, or the setup of the instrument at a new location. In order to obtain chromatograms, it is necessary to connect the data acquisition (DAQ) system. Connect the DAQ system to channel 1 (pin 1: ground; pin 2: positive) of the Model 55 C analog voltage output to obtain chromatograms. In the initial test, the DAQ system's sampling rate should be set to at least 10 Hz.
 - 9.9.2. Test chromatograms should include a blank, a zero air, and a span mix analysis, as follows:
 - 9.9.2.1. Blank: Disconnect the GSS line from the "SAMPLE INLET" fitting, turn off the pump, and run a series of five or more analysis cycles to inject and back-flush the carrier gas. During the first few cycles, the instrument may have methane and non-methane readings, but the chromatograms should appear flat after 2-3 cycles.
 - 9.9.2.2. Zero air: Attach a source of hydrocarbon-free air to the "SAMPLE INLET" fitting. Turn on the pump, then run a series of 5-7 analysis cycles. Be sure to include a vent, or atmospheric pressure dump, in the sampling system to prevent pressurizing the inlet. After a few cycles, the methane and non-methane peaks will go away, and only air peak remains. (Note: Many zero airs do not completely remove methane, thus it is normal to have a small methane peak near the air peak (oxygen peak)).
 - 9.9.2.3. Span gas: The mixture containing approximately 2 ppm each of methane and propane will provide a good test. The span gas should be attached to the "SAMPLE INLET" fitting, and the vent or atmospheric pressure dump should be included to prevent pressurizing the inlet.

9.9.3. Ideally, all test chromatograms should look similar, except for the presence or absence of peaks for methane, NMHCs, and oxygen (Fig. 1). Although spikes occurring at the time of injection and backflush are normal, the baseline should recover within 5 s and should otherwise appear flat and relatively noise free throughout the sample run. If test chromatograms do not meet these criteria, consult the troubleshooting guide in Chapter 5 of the instrument manual.

9.10. Operation

9.10.1. Establish the operating parameters using the menu-driven software, which can be accessed with the front panel or by using RS232 communication system.

9.10.2. The instrument has 6 modes: RUN, MENU, WARM-UP, CALIBRATION, CAL-CHECK, and MANUAL OPERATION.

9.10.2.1. RUN: RUN mode is the normal operation mode, in which the main display shows the analytical results of the most recent analysis cycle. Continuous or single-run operation can be selected from the software's "Run Parameters" menu. In single run, after one analysis, the machine stops. In continuous operation, the instrument automatically performs a series of measurements (at about 70 s per sample) until the user interrupts the run. Analysis may be interrupted by pressing the "MENU" button during a run cycle; at this point, "CANCELED" is displayed in the upper left corner of the main display screen.

9.10.2.2. MENU: Enter the MENU mode by pressing the "MENU" button. In the MENU mode, the instrument software can be accessed from the front panel, but the analytical cycle is stopped.

9.10.2.2.1. Do not press the "MENU" key during the measurement, as this will result in loss of data.

9.10.2.3. WARM-UP: From a cold start, the machine enters the WARM-UP mode until both ovens have stabilized at normal operating temperature. Press the "MENU" key to enter the operational parameters while the instrument is in this mode (Step 9.2.4). The total warm time is about 90 min.

9.10.2.4. CALIBRATION: The Model 55C can be switched into the CALIBRATION mode manually through the menu system, or automatically based on a user-defined calibration setup.

9.10.2.5. CALIBRATION CHECK: This mode is used to check the validity of the current calibration, and is accessed from the "CALIBRATION" menu. In a calibration check, the measurement results are compared to the expected results, but the instrument calibration factors are not adjusted.

9.10.2.6. MANUAL OPERATON: This mode is used in diagnostics and optimization. It is accessed through menu system. In this mode, the FID signal is shown on the fourth line of the main display, but no peak measurements are made.

9.10.3. Main Menu

9.10.3.1. Run Parameters

9.10.3.1.1. Operation mode: Set the operation mode to "Continuous", rather than "Single Measurement". In the latter mode, the instrument stops after each run and waits until the "RUN" button is pressed. Since it is being supplied with sample from the GSS, the former is the appropriate mode.

- 9.10.3.1.2. Instrument range: Used to set the measurement range for both the methane and non-methane channels. The two channels are adjusted independently and may be set to 0-20, 0-200 and 0-2000 ppm. Set AirDAC ranges for methane and non-methane hydrocarbons to match the instrument's ranges.
 - 9.10.3.1.3. Recorder range: These are independent of the instrument range, and are continuously adjustable between 1 and 2000 ppm. The output voltage range for each analog signal is also adjustable, and may be set to any one of four different values.
 - 9.10.3.1.4. Concentration alarms: Used to set the concentration values (in ppm) for methane and non-methane above which the instrument will trigger an alarm. The alarm system can turn on the internal speaker, and also includes a contact closure available on the rear panel.
 - 9.10.3.1.5. Sampling time: For manual operation, the sampling time controls the pause time between pressing the RUN button and the sample injection. For continuous operation, the sampling time controls the pause between measurement cycles. Enter a value between 4 to 999 s that agrees with the sampling time specified in the QAPP or Site Monitoring Plan.
 - 9.10.3.1.6. Response factor: Used to allow the user to set a multiplier that is automatically applied to the NMHC measurement before it is displayed or sent to the analog output. For most applications including measurements at CAFOs, set the response factor at 1.0.
 - 9.10.3.1.7. Date and time: Use this feature to set the instrument's internal clock and calendar.
- 9.10.3.2. Calibration parameters
- 9.10.3.2.1. Gas concentration: Used to input the concentrations of methane and non-methane hydrocarbons in the calibration mixture.
 - 9.10.3.2.2. Auto calibration: Allows user to determine whether or not automatic calibrations will be performed. Four options are available to choose.
 - 9.10.3.2.3. Auto calibration check: Allows the operator to program the instrument to perform automatic span checks at some predetermined interval. Four options are available.
 - 9.10.3.2.4. Auto verification: In most cases, set the auto verification to YES. This allows the instrument to verify a new calibration by automatically running a span check. Calibration cycles are repeated until two consecutive analyses agree to within 2%. If calibration repeatability is not achieved within 5 cycles, an error message and service alarm are generated, and the instrument continues to function using the last valid calibration. Data collected during consecutive cycles will be compared to determine calibration repeatability.
- 9.10.3.3. Calibration
- 9.10.3.3.1. Manual calibration: In this condition, the calibration solenoid opens, and the sampling system is filled with span gas. A complete measurement is then performed.
 - 9.10.3.3.2. Manual calibration check: Used to check repeatability or accuracy of current calibration.

9.10.3.4. Utilities

- 9.10.3.4.1. Temperature: Used to display the FID temperature, the base temperature inside each oven, and the air temperature in the column oven.
- 9.10.3.4.2. Pressures: Used to display the pressure of current air, fuel, and carrier gas (in psi).
- 9.10.3.4.3. Ignite flame: When ignite flame is selected, the system can perform 7 attempts to light the flame; if the flame fails to light, an error message and fault alarm are displayed.
- 9.10.3.4.4. Ignite status: Indicates whether the flame is lit or not.
- 9.10.3.4.5. Clear service alarm: Selecting this menu option clears, or shuts off, all service alarms.
- 9.10.3.4.6. Speaker switch: Allows user to enable or disable the internal buzzer which is located on the mother board.
- 9.10.3.4.7. Column heater: Allows user to enable or disable the column heater.
- 9.10.3.4.8. Detector heater: Allows user to enable or disable the detector heater.

9.10.3.5. Diagnostics

- 9.10.3.5.1. Calibration: These items describe the most recent calibration, and are updated only at the end of a successful calibration cycle. Some of the calibration diagnostics are lost from memory when the instrument is turned off.
- 9.10.3.5.2. Run-time: These describe the FID operation, and present chromatographic data – such as the peak height and retention time - for the most recent analysis. Run-time diagnostics are updated at the end of each analysis, and are retained when the Model 55C is shut off.
- 9.10.3.5.3. System: System diagnostics are updated only in the event of a system failure (such as a flame out) and during the system test that occurs during power-up. System diagnostics include information such as the time of the most recent power-up and the number of flameouts that have occurred.
- 9.10.3.5.4. Set oven temperature: Allows the operator to adjust the set points for the detector and column oven heaters. The permitted range for the detector oven is 50 to 200°C, and the range for the column oven it is 50 to 150°C. These are set at the factory and should not be changed without consulting the TEI service department.
- 9.10.3.5.5. Open fuel valve: Allows user to manually open fuel valve while flame is out. This feature is useful for measuring the fuel flow rate, and for blowing the air out of the fuel system.
- 9.10.3.5.6. Calibration DACs: Allows the user to calibrate the analog outputs on the rear panel of the instrument.
- 9.10.3.5.7. Condition column: Selecting this menu option triggers a column conditioning sequence. This is useful for cleaning a new column or an old column which shows contamination.
- 9.10.3.5.8. Instrument ID: Allows user to adjust the software used by the RS-232 system. The identifier must be included in all RS232 commands, as described in Appendix B of the instrument manual.

9.10.3.6. Windows menu

9.10.3.6.1. Normally not accessed and not available. It may be activated by tuning DIP switch number 1 on the mother board on the “ON” position. When it is activated, it allows the valve actuator and data collection timing to be modified. Do not change any item listed under the Windows menu without first consulting the TEI service department (Section 13.1).

9.10.3.7. Crucial settings

9.10.3.7.1. Used this menu to modify the integration method, or to adjust the calibration of pressure sensors and/or the FID. It can be activated the same way as the Windows menu. Do not change any item listed under the Windows menu without first consulting the TEI service department (Section 13.1).

9.11. Troubleshooting

9.11.1. Troubleshooting guidelines are in Chapter 5 of the instrument manual; servicing instructions are contained in Chapter 6.

9.12. Data acquisition, calculations & data reduction requirements

9.12.1. A 4-20 mA analog output is monitored by AirDAC.

9.12.2. Report data with 3 significant digits.

9.12.3. Follow SOP B4 to correct gas concentrations for calibration and air density.

9.13. Computer hardware & software

9.13.1. AirDAC (SOP B2)

9.13.2. ARDIS (SOP B3)

9.13.3. CAPECAB (SOP B6)

10. Data and Records Management

10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record with a bound record book designated for the method, which contains copies of the electronic record.

10.2. Manage all data according to SOPB5.

10.3. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes. Overstrike all errors in writing with a single line. Initial and date all such corrections.

11. Quality Control and Quality Assurance

11.1. Conduct a Gas Analyzer Calibration at the frequency specified in the QAPP for the particular project.

11.1.1. The Model 55C uses a two-point calibration that incorporates a zero and a span. Zero readings are taken at times when no hydrocarbons are expected to elute from column during the span gas calibration. Calibration of the Model 55C is to be performed daily, according to the following procedure.

11.1.2. Enter the span gas concentrations for the methane and non-methane channels in the “Calibration Parameters” menu.

- 11.1.2.1. To avoid possible interference from oxygen, the methane span concentration must be ≥ 1.5 ppm.
- 11.1.2.2. To obtain good calibration repeatability, the NMHC concentration in the span gas must be > 0.05 ppm.
- 11.1.3. Select either the “Automatic Calibration” or “Calibration Check” option.
 - 11.1.3.1. Set the “Auto Verification” option. For most applications, it should be set to “ON”.
 - 11.1.3.2. Return to the Main Menu and select “Calibration”.
 - 11.1.3.3. Ensure that the calibration gas cylinder is turned on. If the internal calibration solenoid is being used to control the gas flow, adjust the regulator pressure to about 15 psi.
 - 11.1.3.3.1. If a span gas cylinder is connected directly to the instrument, an atmospheric pressure dump, or by-pass must be included to prevent pressuring the sample inlet.
 - 11.1.3.3.2. When using a direct connection to a span cylinder, there must be an excess flow so that the span gas will not be diluted by ambient air or sample.
 - 11.1.3.3.3. If using the internal pump, calibration gases must not be forced into the instrument at elevated pressure.
- 11.1.4. Select “Manual Calibration” to initiate the calibration.
- 11.1.5. During the initial calibration, check to be sure that the span gas flow rate is sufficient to flood the sampling line and prevent ambient air or sample air from being pulled into the instrument.
- 11.1.6. As a general rule, the instrument will be accurate to within 2% at sample concentrations ranging from 20% to 120% of the span concentrations.
- 11.2. The calibration gas should be certified using EPA protocol (1% certification).
- 11.3. Measure the gas analyzer inlet flow rate every six months.
- 11.4. Perform leak check in sample line and flow components and fittings every six months.
- 11.5. The instrument’s guaranteed precision is 2% of reading, and its accuracy is 2% of span.

12. References

- 12.1. Model 55C Manual. 2002. Model 55C Direct Methane, Non-Methane Analyzer Instruction Manual. Thermo Electron Corporation, Franklin, PA
- 12.2. SOP B2. 2006. Data Acquisition and Control Software (AirDAC). Standard Operating Procedure B2. Purdue Ag Air Quality Lab.
- 12.3. SOP B3. 2006. Air Data Pre-Processing Software. Standard Operating Procedure B3. Purdue Ag Air Quality Lab.
- 12.4. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 12.5. SOP B6. 2006. Data Processing Software (CAPECAB). Standard Operating Procedure B6. Purdue Ag Air Quality Lab.
- 12.6. SOP G1. 2006. The PAAQL Gas Sampling System. Standard Operating Procedure G1. Purdue Ag Air Quality Lab.

- 12.7. SOP G2. 2006. Compressed Gas Cylinders. Standard Operating Procedure G2. Purdue Ag Air Quality Lab.
- 12.8. SOP G8. 2006. Multi-Point Calibration of Gas Analyzers. Standard Operating Procedure G8. Purdue Ag Air Quality Lab.
- 12.9. SOP G9. 2006. Precision Checks of Gas Analyzers. Standard Operating Procedure G9. Purdue Ag Air Quality Lab.
- 12.10. SOP Q1. 2006. Use of Control Charts for Performance Monitoring of Gas Analyzers and Analytical Instruments. Standard Operating Procedure Q1. Purdue Ag Air Quality Lab.

13. Contact Information

- 13.1. Manufacturer Contact Information:
 - Thermo Environmental Instruments (www.thermoei.com)
 - 8 West Forge Parkway
 - Franklin, MA 02038
 - Tel: (508) 520-0430 ext. 6812
 - Fax: (508) 520-1460
 - Sales: Souphin A. Sithideth, Inside Sales Engineer, e6812, E-Mail: SSithideth@thermoei.com
 - Service: Barry Pepin, Service Engineer, e6908, E-Mail: Bpepin@thermoei.com
- 13.2. PAAQL: odor@purdue.edu, heber@purdue.edu, <http://www.AgAirQuality.com>.

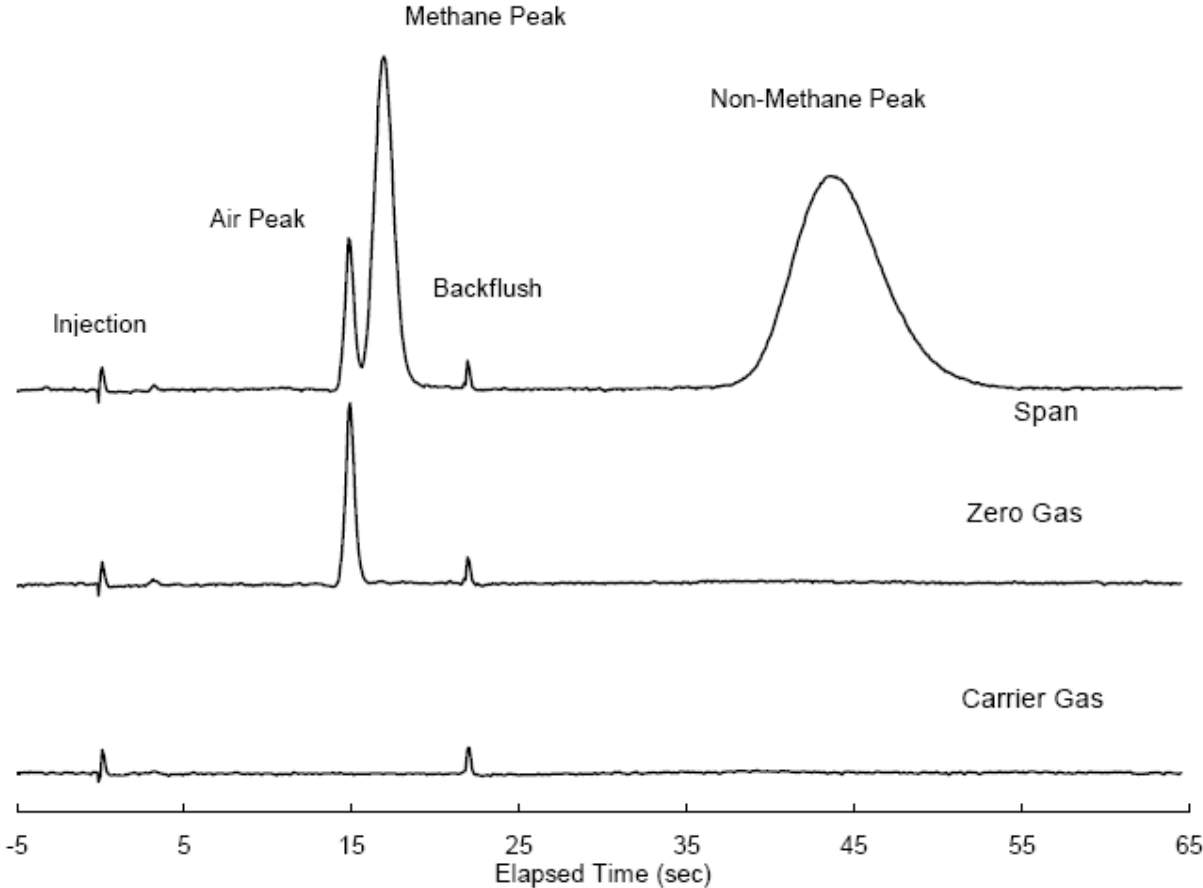


Figure 1. Appearance of normal test chromatograms obtained using the procedures detailed in Section 9.9.

**USE OF THE INNOVA 1412 PHOTOACOUSTIC
MULTI-GAS MONITOR**

Standard Operating Procedure (SOP) G7

USE OF THE INNOVA 1412 PHOTOACOUSTIC MULTI-GAS MONITOR

Standard Operating Procedure (SOP) G7

**Prepared by
Congna Li and Bill W. Bogan**

**Reviewed by
Claude A. Diehl, Jerry Rutherford (California Instruments),
and Albert J. Heber**

Effective Date: November 6, 2006

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions of Terms and Acronyms	5
4. Health and Safety	5
5. Interferences	6
6. Cautions	7
7. Personnel Qualifications.....	7
8. Equipment and Supplies	8
9. Procedure.....	9
10. Data and Records Management	16
11. Quality Control and Quality Assurance	16
12. References.....	17
13. Contact Information	18

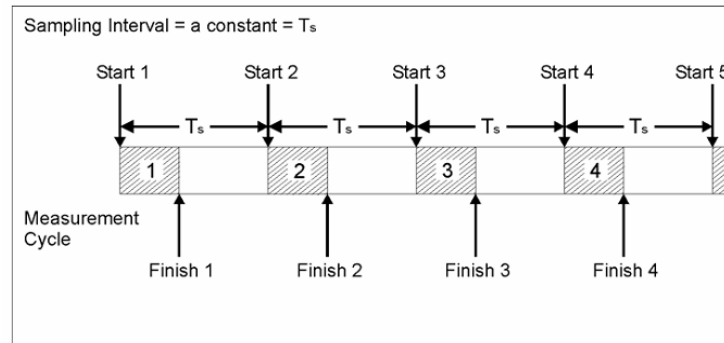
1. Scope and Applicability

- 1.1. The purpose of this procedure is to present the methodology and instrumentation used to automatically and sequentially measure concentrations of various gases at confined animal feeding operations (CAFO), specifically, within the barns and other buildings, and around open sources such as corrals, manure lagoons, or storage basins.
- 1.2. The method described in this SOP is suitable for measurement of any gas which absorbs infrared (IR) radiation. Gas selectivity is achieved through the use of optical filters. Gases which can be measured through this method, and which are relevant to CAFOs, include:
 - 1.2.1. Ammonia (NH₃)
 - 1.2.2. Ethanol/methanol (total alcohols)
 - 1.2.3. Acetic acid
 - 1.2.4. Acetaldehyde
 - 1.2.5. Dimethyl sulfide
 - 1.2.6. Carbon dioxide
 - 1.2.7. Methane
 - 1.2.8. Phenol
 - 1.2.9. Total VOC
- 1.3. The QAPP for a particular project will specify which gases will actually be measured.
- 1.4. This procedure applies only to the INNOVA Model 1412 Photoacoustic Monitor.
 - 1.4.1. The minimum detection limit (MDL) of this instrument is gas-dependent, but is typically in the ppb region. It is often below 100 ppb, with the exception of ammonia, which has a minimum detection limit of 200 ppb.
 - 1.4.2. The range is also gas-specific. The linear range for a given gas is typically from 1 to 10,000 times the MDL.
 - 1.4.2.1. For example, the Model 1412's linear range for ammonia is from 0.2 ppm (the MDL) to 2000 ppm.
 - 1.4.3. The stated precision of the Model 1412 is 1% of measured value.
 - 1.4.4. The instrument's stated response time (97%) to a step change in concentration is 12 s.

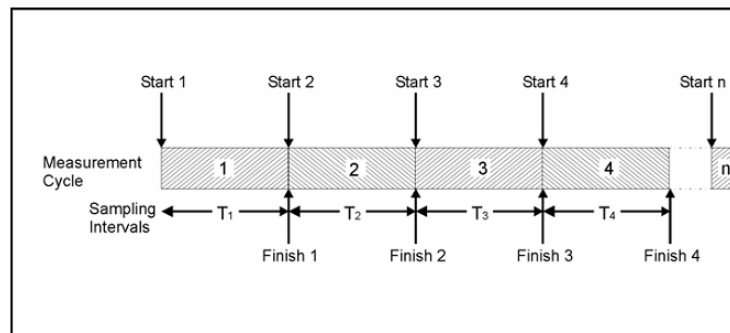
2. Summary of Method

The multi-gas monitor (INNOVA Model 1412, Innova AirTech Instruments, Ballerup, Denmark) is based on the photoacoustic infrared detection method. As shown in the schematic diagram in Fig. 1, a gas sample is drawn at a constant flow rate (in the case of the barns, from the GSS analyzer manifold), and introduced into an acoustic cell where it is exposed to pulsing infrared light of specific wavelength. If the sample contains the gas of interest, it will absorb an amount of infrared light that is proportional to the analyte gas concentration in the sample. When a gas absorbs infrared light, its temperature rises as its molecules increase in kinetic energy; this causes a pressure wave inside the photoacoustic cell. Pressure pulses, which are also proportional to the concentration of the analyte gas, thus correspond to the light pulses, and are detected by two sensitive microphones located inside the chamber. Almost any gas that absorbs infrared light can be measured by this method.

Gas analysis with the Model 1412 can be conducted either using set sampling intervals, or in continuous mode. Sampling interval is a measure of the time between the start of one measurement cycle and the start of the next measurement cycle. Therefore, if a measurement cycle (including readings of all gases being measured) takes 27 s, and the sampling interval is 1 min, then there will be 33 s before the next measurement cycle starts, as follows:



In continuous sampling mode, each measurement cycle is followed immediately by another similar measurement cycle, as below:



In either mode, a measurement cycle consists of the following steps:

- Measurement of the gas sample with each chosen individual optical filter (up to 5)
- Measurement of the gas sample with the water vapor optical filter
- Flushing the chamber & tubing and filling the chamber with the next gas sample

Gas selectivity is achieved through the use of optical filters. The Innova 1412's optical filter carousel can hold 5 optical filters for target gases, plus one for water vapor, thus allowing measurement of up to 5 gases and water vapor in a given air sample. Each individual optical filter rotates in front of the photoacoustic chamber, and pauses for the set sample integration time (SIT) before moving to the next filter. All filters rotate through once every measurement cycle. The MDL is gas-specific and filter wavelength-specific; examples include:

Acetic acid	40 ppb	Total TOC (reference propane)	20 ppb
Ammonia	200 ppb	Methane	100 ppb
Carbon dioxide	1500 ppb	Sulfur dioxide	300 ppb

Measurement accuracy is ensured by the Model 1412's ability to compensate for temperature and pressure fluctuations, water-vapor interference and interference from other IR-absorbing gases that are known to be present. Reliability of measurement results can be ensured by regular self-tests. The analysis time is dependent on the physical properties of the gas of interest, the number of gases being measured, the set-up of the instrument, the gas sampling system, and the sampling location. When a gas or a gas mixture is introduced to the analyzer, the response time can vary from approximately 13 s for one gas (or water vapor) to approximately 150 s if five gases plus water vapor are measured. The following table indicates in more detail the effect of analyzer configuration on analysis time:

Sample integration time, s	1	1	5	5	20
Water vapor measured?	no	yes	no	yes	yes
Number of gases	1	5	1	5	5
Flushing time, s	4	4	Auto (tube 1 m)	Auto (tube 1 m)	Auto (tube 1 m)
Analysis Time, s	~13	~27	~25	~60	~150

3. Definitions of Terms and Acronyms

AirDAC	Data Acquisition and Control software (SOP B2)
Analysis Time	The total time required to measure each gas's response to infrared light (SIT) and to flush the photoacoustic cell.
GSS	Gas sampling system (SOP G1)
IR	Infrared
LAN	Local area network
MDL	Minimum detection limit
NIST	National Institutes of Science and Technology
NMi	Netherlands Measurement Institute
PAAQL	Purdue Agricultural Air Quality Laboratory
PAML	Purdue Applied Meteorology Laboratory
PC	Personal computer
ppb	Parts per billion
ppm	Parts per million
QAPP	Quality Assurance Project Plan
SIT	Sample integration time

4. Health and Safety

- 4.1. Be careful when working with the analyzer's electrical power connection.
- 4.2. The IR source and analysis cell are very hot when the analyzer is operating. Do not attempt to service any internal components without allowing adequate cool-down time.
- 4.3. If used indoors (such as inside the OFIS). Properly vent the analyzer's exhaust to avoid exposure to noxious gases in the sample.

5. Interferences

- 5.1. Water absorbs IR light at most wavelengths, and is nearly always present in air samples. Thus, water will essentially always contribute to the total acoustic signal. The Model 1412 is permanently fitted with a special water-selective filter, which enables it to compensate for this interference.
- 5.2. By selecting different filters, this technique can also be used to cross-compensate for known interferents (i.e. gases which are not target analytes, but which are known to absorb IR at the wavelength(s) used to quantify one or more target compounds.
 - 5.2.1. Possible interferent gases relevant to livestock barns, anaerobic treatment lagoons, manure storages, and related facilities (Schmidt et al, 2005; Summers, 2005) include the following (levels are based on detection limits in Innova AirTech Instruments' "Detection Limits Wall Chart" (Reference 12.3) for these gases at the same wavelength used for quantifying the given primary analyte):
 - 5.2.1.1. **Ammonia** can be quantified at either of two wavelengths – 974 μm or 976 μm . The stated detection limit at each of these wavelengths is 200 ppb. Some of these compounds will interfere more at one wavelength (indicated in parentheses) and less (but still possibly significantly) at the other wavelength. If VOC characterization of the site indicates that any of these compounds are present above the indicated thresholds, it may be necessary to compensate for them in order to obtain accurate NH_3 data.
 - 5.2.1.1.1. 2-butanone \geq 500 ppb (976 μm)
 - 5.2.1.1.2. Ethanol \geq 60 ppb (974 μm)
 - 5.2.1.1.3. Ethanolamine \geq 110 ppb (974 μm)
 - 5.2.1.1.4. Methanol \geq 80 ppb (974 μm)
 - 5.2.1.1.5. Phenol \geq 400 ppb (974 μm)
 - 5.2.1.2. **Ethanol/methanol** is optimally measured at 974 μm , with a detection limit of 80 ppb. Interferents at this wavelength include:
 - 5.2.1.2.1. Ammonia \geq 200 ppb
 - 5.2.1.2.2. Ethanolamine \geq 110 ppb
 - 5.2.1.2.3. Phenol \geq 400 ppb
 - 5.2.1.3. **Acetic acid** is optimally measured at 970 μm . Its stated detection limit is 40 ppb. Interferents at this wavelength include:
 - 5.2.1.3.1. Methanol \geq 500 ppb
 - 5.2.1.4. **Acetaldehyde** is optimally measured at 986 μm , with a detection limit of 80 ppb. Interferents at this wavelength include:
 - 5.2.1.4.1. Dimethyl sulfide \geq 400 ppb
 - 5.2.1.4.2. Dimethylamine \geq 40 ppb
 - 5.2.1.4.3. Formaldehyde \geq 40 ppb
 - 5.2.1.4.4. Trimethylamine \geq 20 ppb
 - 5.2.1.5. **Dimethyl sulfide** is optimally measured at 936 μm . Its detection limit is 600 ppb at this wavelength. Interferents at this wavelength include:
 - 5.2.1.5.1. Methanol \geq 80 ppb
 - 5.2.1.6. No obvious relevant interferents for **CO₂** (quantified at 983 μm) are evident. The stated detection limit for **CO₂** is 3.4 ppm

- 5.2.1.7. **Methane** can be quantitated at 968 μm or 969 μm ; however, the former of these is much more sensitive to interference from water vapor. When methane is quantified at 969 μm , it has a detection limit of 400 ppb, with the following possible interferents:
- 5.2.1.7.1. $\text{H}_2\text{S} \geq 14$ ppm
 - 5.2.1.7.2. Acetone ≥ 70 ppb
- 5.2.1.8. **Phenol** is optimally measured at 971 μm . Its detection limit is 8 ppb. Interferents at this wavelength include:
- 5.2.1.8.1. m-cresol ≥ 50 ppb
 - 5.2.1.8.2. Dimethylamine ≥ 200 ppb
 - 5.2.1.8.3. Methyl methacrylate ≥ 20 ppb
 - 5.2.1.8.4. $\text{SO}_2 \geq 400$ ppb
 - 5.2.1.8.5. Trimethylamine ≥ 200 ppb
- 5.2.1.9. Total VOC is best measured at 987 μm , with a detection limit of 20 ppb. Since it is intended to measure all volatile organic hydrocarbons, no individual hydrocarbons can be considered interferents. Some oxygenated VOC will absorb at this wavelength; others will not. Ammonia and H_2S have negligible absorptions at 987 μm .
- 5.3. Certain gases require humidification in order to generate accurate calibration readings. This is needed to speed the relaxation (energy release) of the analyte molecules after they are excited by the IR light, by mediating the dispensation of energy to bulk fluid (gas). Without humidification, there will be significant downward drifting of the reading of the calibration gas, leading to inaccurate readings. An inline Nafion-based humidifier (Section 8.5) must be used (Section 9.4.3.3.1.3) with these gases.
- 5.3.1. Humidification is required for any calibration or span check with CO_2 .
 - 5.3.2. Humidification is required for methane if air is used as the diluent.

6. Cautions

- 6.1. When installing the RS-232 cable to the back of the gas analyzer monitor, ensure that both the monitor and the PC are switched off at the mains. Failure to do so may result in equipment being damaged.
- 6.2. Blown fuses in the monitor can indicate a serious fault in the analyzer; therefore, it is advisable to contact the Innova representative before changing the fuse.
- 6.3. Do not pressurize the instrument above 250 Pa (1 in. of water).
- 6.4. Avoid water condensation in the analyzer.
- 6.5. Do not allow any liquid to enter the analyzer's photoacoustic chamber.

7. Personnel Qualifications

- 7.1. Personnel should be trained in the use of the analyzer before initiating the procedure. Training requires about three hours.
- 7.2. Each analyst must read and understand the entire analyzer manual and this SOP before operating the Model 1412.

- 7.3. Personnel should also be trained to interpret the analyzer output signal, converted concentration data, and correction of concentrations based on calibration.

8. Equipment and Supplies

- 8.1. Multi-gas Photoacoustic Monitor (Model 1412, Innova AirTech Instruments, Ballerup, Denmark), with appropriate optical filters (as defined in the particular project's QAPP):
 - 8.1.1. UA0976 or 974 for ammonia
 - 8.1.2. UA0974 for ethanol/methanol
 - 8.1.3. UA0970 (or UA0971) for acetic acid
 - 8.1.4. UA0986 for acetaldehyde
 - 8.1.5. UA0936 for dimethyl sulfide
 - 8.1.6. UA0983 for carbon dioxide
 - 8.1.7. UA0968 for methane
 - 8.1.8. UA0971 for phenol
 - 8.1.9. UA0985 for nitrous oxide
 - 8.1.10. UA0984 for carbon monoxide
 - 8.1.11. UA0987 for total VOC (general hydrocarbon filter)
- 8.2. Rack and rack mounts
- 8.3. Data acquisition and control
 - 8.3.1. Barn applications: PAAQL data acquisition and control hardware (SOP B1), and AirDAC (SOP B2) and proprietary custom software to allow communication between INNOVA and AirDAC (Nexus Solutions, London, Ontario, Canada)
 - 8.3.2. Open-source applications: LAN Server computer in open-source trailer (SOP U5 or U7 for lagoon or corral sites, respectively), and LabVIEW software (National Instruments Corporation, Austin, TX)
- 8.4. Mounting post and post driver (for open-source applications – SOP U5 or U7)
- 8.5. Nafion humidifier (California Analytical Instruments Part # CAI-110-03-34)
- 8.6. Keep the following spare parts at each remote site and in central stocks at PAAQL and PAML:
 - 8.6.1. Threaded nuts (2x YM 0652)
 - 8.6.2. Swagelok stainless steel ¼" nuts and ferrules
 - 8.6.3. Gas Monitoring Software 7304 for Photoacoustic Multi-gas monitors 1412
 - 8.6.4. External filters
 - 8.6.4.1. (Recommended) stainless steel filter frit (10 µm, 25 mm)
 - 8.6.4.2. External fine air filters (CAII-F01) 100/pkg. (Millipore: Mitex Membrane Filter, PTFE, hydrophobic, 10 µm, 25 mm, white, plain; catalog number: LCW02500)
 - 8.6.5. Cables
 - 8.6.5.1. Modem cable (9 pin)
 - 8.6.5.2. RS-232 null modem cable (25 pin female/9 pin male)
 - 8.6.5.3. Power cable (cord) for the analyzer
 - 8.6.6. Tubing
 - 8.6.6.1. Teflon tubing (¼" OD)
 - 8.6.6.2. UD 5037 Nafion tubing

- 8.6.7. Spare slow-blow (T) fuses with a rating of 2.5 A, 250 VAC, 5x20 mm, UL-approved (Innova Order Number: VF0087A)
- 8.6.8. Instruction manual, menu tree, and field guide for the Innova 1412 Photoacoustic Monitor
- 8.7. Keep the following spare parts in central stocks at PAAQL and PAML:
 - 8.7.1. Optical filter locking springs (8x DL 3322)

9. Procedure

- 9.1. Installation
 - 9.1.1. Check analyzer carefully to assure it is free of visible defects or damage.
 - 9.1.2. Mounting options
 - 9.1.2.1. For barn applications, securely mount the analyzer horizontally on the instrument rack in the OFIS.
 - 9.1.2.2. For open-source applications, mount the instrument securely to a mounting post at a specified location downwind of the lagoon or other open source which is being monitored. See the Site Monitoring Plan to locate the exact location.
 - 9.1.3. Install the gas monitoring software 7304 for the control of photoacoustic gas monitors 1412 and 1314, if it is not already installed.
 - 9.1.4. Connect the gas analyzer monitor to the data acquisition PC.
 - 9.1.4.1. Connect the 25-pin female cable (RS-232) to the RS-232 port on the Innova 1412. Connect the 9-pin modem cable to the RS-232 cable from the Innova 1412. Connect the null modem cable into the COM 1 port of the DAC computer.
 - 9.1.4.2. Leave sufficient extra length to the tubing and cables such that the instrument rack can be moved a few feet to get access to the back of the analyzer.
 - 9.1.5. Connect tubing to the Innova 1412
 - 9.1.5.1. Connect the analyzer inlet to the gas source (e.g. the GSS analyzer manifold for barn applications) with the minimum length of ¼" OD Teflon tube.
 - 9.1.5.2. Connect the analyzer exhaust with a 3" length of 4-mm tube that is connected to ¼" ID PVC or other tubing. Connect the exhaust tube from the analyzer to the air exhaust of the OFIS.
 - 9.1.6. Power up the analyzer, turn on its internal pump, switch to measurement mode and allow the analyzer to warm up for a minimum of 60 – 90 min before analysis. This is necessary to warm up the analysis cell, as conditions within the cell tend to stabilize more quickly (allowing stable readings) once the temperature inside the analysis cell is 15°C above the ambient room temperature.
- 9.2. Setting up Communication Parameters between the Model 1412 and PC
 - 9.2.1. Using the push buttons on the front of the monitor and following the operation manual, set the communication parameters for the serial interface as follows:

Baud rate	9600
Stop bits	1
Data bits	7
Parity	Even
Hardwire mode	Leased line
Handshake type	Hardwire

- 9.2.2. Using the push buttons on the front of the monitor and follow the operation manual, set the text line terminator, print data log and print error log as follows to prevent communication errors:

Text line Terminator	CR-LF
Print Data Log	NO
Print Error Log	NO

- 9.2.3. Select the PC communication port
- 9.2.3.1. Start any one of the gas monitoring software 7304 options: Online, Offline, or Calibration.
 - 9.2.3.2. Pull down the Task menu. Click on Communication. The analyzer can use COM port 1 through COM port 4.
 - 9.2.3.3. Click on the radio push-button next to the correct port name. Refer to the PC manual if not sure which port the cable is connected to. COM Port 1 is used most of the time.
- 9.3. Set up and start a Monitoring Task using the gas monitor software 7304, and set the instrument parameters according to Table 1.
- 9.3.1. Measurement parameter set-up
- 9.3.1.1. Pull down the Task menu. Click on New to create a new database, or click on Open to use an existing database.
 - 9.3.1.2. For a new database, click in the “Enter Name For” field of the database window and type in a desired name. For existing databases, click on the desired database name.
 - 9.3.1.3. Click OK.
 - 9.3.1.4. Pull down the Sequence menu. Click on Settings and the Setup window appears.
 - 9.3.1.5. Click on the Sampling tab.
 - 9.3.1.6. Click on the radio-button for the desired sampling mode. If Sampling Interval is selected, set the time to the correct value.
 - 9.3.1.7. Click on the radio-button for the desired flushing mode.
 - 9.3.1.7.1. If Auto is selected, set the correct tube length.
 - 9.3.1.7.2. If Fixed Time is selected, set the desired chamber flushing time and the tube flushing time (recommended value: chamber flushing time, 8 s; tube flushing time, 3 s).
 - 9.3.1.8. If water vapor and cross interference are required, check the appropriate box.
 - 9.3.1.9. If average values are to be displayed on the gas monitor screen, click in the Average check box, and set the interval to the correct value.
 - 9.3.1.10. Click on the Gas tab.
 - 9.3.1.11. Click in the check box to the left of the desired filter.
 - 9.3.1.12. Click in the field to the right of the selected filter and select the correct gas.
 - 9.3.1.13. Repeat the above two steps until all the filters which are required for the measurements are selected and the correct names are displayed.
 - 9.3.1.14. Click in the Sample Integration Time field and select the desired option.
 - 9.3.1.15. Click on the Alarm tab and check to make sure that alarms are set to default.
 - 9.3.1.16. Click on OK to complete the system setup.

Table 1. Appropriate settings for various instrument parameters for the INNOVA Model 1412.

Instrument Parameters	
Units	
Concentration Unit:	ppm
Length Unit:	ft
Temperature Unit:	°C
Pressure Unit:	mm Hg
Humidity Unit:	Tdew
Normalization Temperature	20.0°C
Sampling Mode	
Sample Continuously	Yes
Sampling Interval (If selected)*	1 min (barns), 200 s (open-source)
Flushing	
Auto	No
Tube Length	10 ft (barns), 0.5 ft (open-source)
Fixed Time	Yes
Chamber	8 s (barns), 13 s (open-source)
Tube	3 s
Compensation	
Water Interference	Yes
Cross Interference	Yes
Gas Monitor Display	
Average	No
Time (HH:MM)	00:10
Current Air Pressure	
Pressure	Measured by Instrument
Memory Use	
Store History	Yes
Gas Setup	
Gas	S.I.T.
Ammonia	2 s
Ethanol / Methanol	1 s
Acetic Acid	2 s
Acetaldehyde	2 s
Dimethyl Sulfide	1 s
Carbon Dioxide	1 s
Methane	1 s
Phenol	2-5 s
Total VOC	1s
Alarms	
All gases are set to "Default"	No Alarms Set

* Note: If "Sampling Interval" Mode is selected, set "Sample Continuously" to "No".

- 9.3.2. Start a monitoring task
 - 9.3.2.1. Pull down the Sequence menu and click on Start.
 - 9.3.2.2. In the “Enter Sequence Description” field, type in the name of the monitoring sequence. If no name is entered, the default name “Sequence 1” is used.
 - 9.3.2.3. To start the measurement immediately, click on the Now radio-button in the Start Time group.
 - 9.3.2.4. To delay the start, click on the Start At: radio-button, and define the start time in the Start Time group.
 - 9.3.2.5. To manually stop the measurement later, click on the None radio-button in the Stop Time group.
 - 9.3.2.6. To automatically stop the measurement by defining a stop time, click on the Stop at: radio-button in the Stop Time group and enter the time to stop.
 - 9.3.2.7. Press OK to start the monitoring task.
- 9.3.3. Stop a monitoring task
 - 9.3.3.1. If no pre-set monitoring period is defined, pull down the Sequence menu and click on Stop.
- 9.3.4. Displaying measurement results while monitoring
 - 9.3.4.1. Using the Online program, measurement data are displayed on screen as soon as they are available from the monitor. The data can be displayed in both a graphic window and a numeric window, simultaneously. Refer to the manual for detailed procedures for defining the graphic window and numeric window.
- 9.3.5. Displaying measurement results from a stored monitoring task
 - 9.3.5.1. Click on the Presentation program, and pull down the Task menu. Click on Open to view an existing database.
 - 9.3.5.2. In the database window, click on the desired database name.
 - 9.3.5.3. Click on OK. A window opens showing all the measurement values for the complete monitoring task. Refer to the manual for detailed procedures for defining the graphic window and numeric window.
- 9.3.6. Export measurement data as comma-delimited ASCII files
 - 9.3.6.1. Click on the Presentation program. Open the database to be exported.
 - 9.3.6.2. Pull down the Task menu and click on Export.
 - 9.3.6.3. Use the standard Windows browser, which appears on screen, to define the destination for the exported data.
 - 9.3.6.4. Type in the desired file name. All files have a .TXT extension.
 - 9.3.6.5. Click on Save.
 - 9.3.6.6. Use Microsoft Excel to open and process the saved file.
- 9.4. Gas Analyzer Calibration
 - 9.4.1. The exact composition for each reference calibration gas is dependent upon the gases being monitored in a given project, and the concentrations expected at the site. Calibration gas compositions for a particular project will be specified in the QAPP for that project.
 - 9.4.2. Calibrate Innova 1412 following SOPs G8 (multipoint calibrations) and G9 (zero/precision checks).

9.4.3. Calibrate the optical filters.

9.4.3.1. Calibrate the optical filters with the following steps: zero point calibration, humidity interference calibration, span calibration of CO₂ (if that filter is in the instrument), and span calibration of any other gases after the instrument has been dried with zero air or a clean dry source of nitrogen.

9.4.3.2. Before calibrating the optical filters:

9.4.3.2.1. Warm up the monitor for a period of 60-90 min in the measurement mode before a calibration task is started to reduce the time required for calibration.

9.4.3.2.2. Detach the air inlet tube from the existing sampling manifold. Close the gas sampling manifold with a plug.

9.4.3.2.3. Obtain a print-out of calibration data in the monitor by using the "Print Current Calibration" function. This will compare the "old" calibration factors with the "new" ones.

9.4.3.2.4. Check the calibration of each installed optical filter following the procedures described in the manual.

9.4.3.2.5. Verify that the installed optical filters have been entered correctly in the active set-up of the monitor.

9.4.3.3. Basic setup for calibration

9.4.3.3.1. Set up the calibration equipment according to Fig. 2. Connect three different lengths of Teflon tubing to a "Y"-piece.

9.4.3.3.1.1. Connect the tube attached to point 1 to the analyzer's air inlet.

9.4.3.3.1.2. Connect the tube attached to point 2 to a gas flow meter.

9.4.3.3.1.3. Connect the tube attached to point 3 to the cylinder of gas which is to be used during calibration. This connection should be direct if humidity is not to be included, or through a water bath (as shown in Fig. 3) if humidity interference calibration is being conducted. If the calibration is being done for CO₂ or methane, use a Teflon tube which has been cut into two segments, and with a Nafion humidifier inserted into it using the tubing couplers provided.

9.4.3.4. Preparation for humidity interference, zero, or span calibration

9.4.3.4.1. Verify the settings of the Model 1412's communication parameters are set to the values described in Section 9.2.1.

9.4.3.4.2. Make sure the text line terminator, print data log and print error log are set to the values described in Section 9.2.2.

9.4.3.4.3. Open the "Calibration" program and create a new calibration task.

9.4.3.5. Zero gas calibration

9.4.3.5.1. Close the regulator valve on the zero gas cylinder.

9.4.3.5.2. Open the main valve on the zero gas cylinder.

9.4.3.5.3. Open the "Calibration" program and choose "New" to create a new task.

9.4.3.5.4. Type in the desired task name and click "OK".

9.4.3.5.5. Pull down the "Sequence" menu and click "Settings".

9.4.3.5.6. Click on the "Calibration" tab, if it is not already at the front. Select the "Zero point" button.

9.4.3.5.7. Click on the "Gas" tab, click in the "Sample Integration Time" field, and make sure that SIT is set to the same as that used for monitoring (Section 9.3).

- 9.4.3.5.8. Click on the Sampling tab and set the flushing time. The flushing type and values should be set to the same as those used when monitoring. Click OK.
- 9.4.3.5.9. Insert the 1/4"-OD tubing from the zero gas cylinder into the flow meter mounted on the side of the instrument rack. Open the regulator valve to allow gas flow. Adjust regulator valve until vent airflow is about 2.5 L/min. Use the flow meter to measure the flow rate.
- 9.4.3.5.10. Pull down the "Sequence" menu, and click "Start". Record time and analyzer display in field notes.
- 9.4.3.5.11. After display has stabilized (typically 10 to 15 min), pull down the "Sequence" menu and click "Stop".
- 9.4.3.5.12. Close regulator and remove tubing from the zero gas cylinder.
- 9.4.3.6. Humidity interference calibration
 - 9.4.3.6.1. Run the Teflon tube from the zero gas cylinder into a temperature-controlled water bath. The outlet tube from the water bath should flow into a separate, empty flask, which acts as a safety trap. The outlet line from this flask then enters the "Y" connector, and is split between the flow meter and monitor. See Fig. 3 for a diagram of this setup.
 - 9.4.3.6.1.1. The temperature of the water bath and the safety flask should be at least 2°C below the ambient temperature of the room.
 - 9.4.3.6.2. Pull down the "Sequence" menu and click "Settings".
 - 9.4.3.6.3. Click on the "Calibration" tab, if it is not already at the front.
 - 9.4.3.6.4. Click to select "Humidity Calibration" radio-button.
 - 9.4.3.6.5. Only select Humidity Interference Calibration (All Filters). Do not select Perform Water Vapor Span Calibration.
 - 9.4.3.6.6. Click on the "Gas" tab, click in the "Sample Integration Time" field, and select the desired time (Fast 1 s).
 - 9.4.3.6.7. Click "OK".
 - 9.4.3.6.8. Open the regulator valve to allow gas flow through the water bath and safety flask (Do not allow any liquid into the instrument).
 - 9.4.3.6.9. Adjust regulator valve until vent airflow is about 2.5 L/min using a flow meter to measure the flow rate.
 - 9.4.3.6.10. Pull down the "Sequence" menu and click "Start".
 - 9.4.3.6.11. Allow the readout to stabilize (typically 10 to 15 min). Record time and analyzer display in field notes.
 - 9.4.3.6.12. Pull down the "Sequence" menu; click on "Stop".
 - 9.4.3.6.13. Close regulator and main valve and remove tubing from the zero gas cylinder.
- 9.4.3.7. Span gas calibration of CO₂ (if applicable)
 - 9.4.3.7.1. Pull down the "Sequence" menu, and click "Settings".
 - 9.4.3.7.2. Click on the "Calibration" tab, if it is not already at the front. Select the "Gas Span Calibration" button.
 - 9.4.3.7.3. Click in the "Active Filter" field, select "Carbon Dioxide", or other gas as appropriate. Check "Perform Cross Interference Calibration". Input the concentration of the analyte in the gas which is being used.
 - 9.4.3.7.4. Click on the "Gas" tab, click in the "Sample Integration Time" field, and select the desired time (the same as that used when monitoring). Click "OK".

- 9.4.3.7.5. Insert the ¼" OD tubing from the gas cylinder into the flow meter mounted on the side of the instrument rack. Open the regulator valve to allow gas flow. Adjust regulator valve until vent airflow is about 2.5 L/min, using the flow meter to measure the flow rate.
- 9.4.3.7.6. Pull down the "Sequence" menu, and click "Start". Record time and analyzer display in field notes.
- 9.4.3.7.7. After the display has stabilized (typically 10 to 15 min), pull down the "Sequence" menu and click "Stop".
- 9.4.3.7.8. Close regulator and remove tubing from the gas cylinder.
- 9.4.3.8. Span gas calibration for other gases
 - 9.4.3.8.1. Set the analyzer to measure water vapor only
 - 9.4.3.8.2. Flow dry nitrogen or CEM zero air through the analyzer until the water vapor measurement is reading -58 to -60 for Tdew°C.
 - 9.4.3.8.3. Pull down the "Sequence" menu, and click "Settings".
 - 9.4.3.8.4. Click on the "Calibration" tab, if it is not already at the front. Select the "Gas Span Calibration" button.
 - 9.4.3.8.5. Click in the "Active Filter" field, select "Ammonia", or other gas as appropriate. Check "Perform Cross Interference Calibration". Input the concentration of the analyte in the gas which is being used.
 - 9.4.3.8.6. Click on the "Gas" tab, click in the "Sample Integration Time" field, and select the desired time (have to be the same as those used when monitoring). Click "OK".
 - 9.4.3.8.7. Insert the ¼" OD tubing from the gas cylinder into the flow meter mounted on the side of the instrument rack. Open the regulator valve to allow gas flow. Adjust regulator valve until vent airflow is about 2.5 L/min using a flow meter to measure the flow rate.
 - 9.4.3.8.8. Pull down the "Sequence" menu, and click "Start". Record time and analyzer display in field notes.
 - 9.4.3.8.9. After display has stabilized (typically 10 to 15 min), pull down the "Sequence" menu and click "Stop".
 - 9.4.3.8.10. Close regulator and remove tubing from the gas cylinder (Repeat Sections 9.4.3.7.1 through 9.4.3.7.8 for the other gases).
- 9.4.3.9. Calculating and downloading Calibration Factors
 - 9.4.3.9.1. While the raw measurement data are still displayed on the screen, open the "Cursor Values" dialogue box.
 - 9.4.3.9.2. Use two cursors and the statistical data (displayed in the Cursor Values dialogue box) to locate a suitable range of data. All values in the selected interval should be very stable, and the temperature should be above 40°C.
 - 9.4.3.9.3. When the desired region is between the cursors, pull down the "Sequence" menu, and click on "Mark Interval".
 - 9.4.3.9.4. Select and mark all the intervals for every calibration.
 - 9.4.3.9.5. Pull down the "Value" menu, and click on "Calculate". When the calculation(s) is complete, a "Calculation Finished" dialogue box is displayed.
 - 9.4.3.9.6. Pull down the "Values" menu, and click on "Download". The "Download" dialogue box is displayed.

- 9.4.3.9.7. Select the “Zero Point” tab, and set ticks in the appropriate check boxes, depending on which gas (and/or water vapor) calibrations were just run.
- 9.4.3.9.8. Repeat the above two steps in the “Humidity” and “Gas” tabs.
- 9.4.3.9.9. Click “OK” to download the calibration factors to the monitor.
- 9.4.3.10. After calibration is complete, reattach the Model 1412 to the GSS, and close the main valves on all gas cylinders.
- 9.4.4. Data acquisition, calculations & data reduction requirements
 - 9.4.4.1. Report data with three significant digits.
 - 9.4.4.2. Follow SOP B4 to correct gas concentrations for calibration and air density.
- 9.4.5. Computer hardware & software
 - 9.4.5.1. Barn applications:
 - 9.4.5.1.1. AirDAC (SOP B2)
 - 9.4.5.1.2. AirDAC Data Pre-Processing Software (SOP B3)
 - 9.4.5.1.3. CAPECAB (SOP B6)
 - 9.4.5.2. Open-source applications (SOPs D1, U4, and U5/U7):
 - 9.4.5.2.1. LabVIEW
 - 9.4.5.2.2. LAN Server computer

10. Data and Records Management

- 10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement his electronic record with a bound record book designated for the method, which should contain copies of the electronic record.
- 10.2. Manage all data according to SOP B5 or SOP D1 (barn or open-source applications), as appropriate.
- 10.3. Document all data and information (e.g., sample collection method used) on field data sheets, and within site logbooks with permanent ink, or in electronic field notes.
- 10.4. Overstrike all errors in writing with a single line. Initial and date all such corrections.

11. Quality Control and Quality Assurance

- 11.1. Conduct gas analyzer checks according to the QAPP for the particular project. This will include zero gas and one or more reference span concentrations.
- 11.2. Conduct gas analyzer calibration when the precision check is $>\pm 10\%$ of baseline, unless the QAPP for the particular project specifies a different value.
- 11.3. Conduct gas analyzer calibration when the zero check is $>\pm 5\%$ of full scale different than the baseline, unless the QAPP for the particular project specifies a different value.
- 11.4. The calibration gas should be certified using EPA protocol (1% certification), if this grade is available, unless the QAPP for the particular project specifies a different value.
- 11.5. Check the response time every six months, unless the QAPP for the particular project specifies a different value.
- 11.6. Measure the gas analyzer’s inlet flow rate every six months.
- 11.7. Perform leak check in sample line and flow components and fittings every six months by measuring the inlet and exhaust flow rates with a flow meter.

- 11.8. Change the fine air-filter paper in the internal and external air-filtration units monthly. For analyzers that have the stainless steel 10 µm frit for the internal filter, clean the frit monthly. After cleaning or replacing filters, perform a leak check at the inlet sample port and the exhaust port by measuring the inlet and exhaust flow rates with a flow meter, to ensure that no leaks were introduced.
- 11.9. Clean the filter in the ventilation unit monthly.
- 11.10. Record any maintenance in the Instrument Performance Record File's worksheet labeled maintenance.

12. References

- 12.1. Model 1412 Photoacoustic Field Gas-Monitor Product Data. Innova AirTech Instruments. Online at www.innova.dk/uploads/media/Product_Data_1412_PW.pdf. Accessed 2/15/2006.
- 12.2. Model 1412 Photoacoustic Field Gas-Monitor Details. Innova AirTech Instruments. Online at http://www.innova.dk/1412_details.gas_monitoring4.0.html. Accessed 2/15/2006.
- 12.3. Innova AirTech Instruments. 2000. Detection Limits Wall Chart. Online at www.innova.dk/uploads/media/Wall_Chart_PW_01.pdf. Accessed 2/15/2006.
- 12.4. Schmidt, C.E., T. Card, and P. Gaffney. 2005. Assessment of reactive organic gases and amines from a Northern California dairy using the USEPA surface emission isolation flux chamber. Presented at the California Air Resources Board Livestock Emissions Symposium. January 26, 2005. Online at <http://www.arb.ca.gov/ag/caf/lersymp.htm>. Accessed 2/17/2006.
- 12.5. Summers, M.D. 2005. Emissions from California poultry production. Presented at the California Air Resources Board Livestock Emissions Symposium. January 26, 2005. Online at <http://www.arb.ca.gov/ag/caf/lersymp.htm>. Accessed 2/17/2006.
- 12.6. SOP B2. 2006. Data Acquisition and Control Software (AirDAC). Standard Operating Procedure B2. Purdue Ag Air Quality Lab.
- 12.7. SOP B3. 2006. Air Data Pre-Processing Software. Standard Operating Procedure B3. Purdue Ag Air Quality Lab.
- 12.8. SOP B4. 2006. Calculation and Reporting of Air Emissions from Barns. Standard Operating Procedure B4. Purdue Ag Air Quality Lab.
- 12.9. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 12.10. SOP B6. 2006. Data Processing Software (CAPECAB). Standard Operating Procedure B6. Purdue Ag Air Quality Lab.
- 12.11. SOP D1. 2006. Management of Open-source, Weather, and Lagoon-characterization Data. Standard Operating Procedure D1. Purdue Applied Meteorology Lab.
- 12.12. SOP G8. 2006. General Calibration of Gas Analyzers. Standard Operating Procedure G8. Purdue Ag Air Quality Lab.
- 12.13. SOP G9. 2006. Zero and Precision Checks of Gas Analyzers. Standard Operating Procedure G9. Purdue Ag Air Quality Lab.

12.14.SOP U5. 2006. The Installation of Open-source Measurement Equipment. Standard Operating Procedure U5. Purdue Applied Meteorology Lab.

12.15.SOP U7. 2006. The Instrument Trailer and Installation of Open-source Measurement Equipment (Corrals). Standard Operating Procedure U7. Purdue Applied Meteorology Lab.

13. Contact Information

13.1. Distributor:

California Analytical Instruments

Tel: (714) 974-5560

Email: info@gasanalyzers.com

13.2. Manufacturer:

Innova AirTech Instruments (www.innova.dk)

Energivej 30, DK-2750 Ballerup, Denmark

Tel.: (+45) 44 20 0100, Fax: (+45) 44 20 0101, innova@innova.dk

13.3. PAAQL: odor@purdue.edu, heber@purdue.edu, <http://www.AgAirQuality.com>.

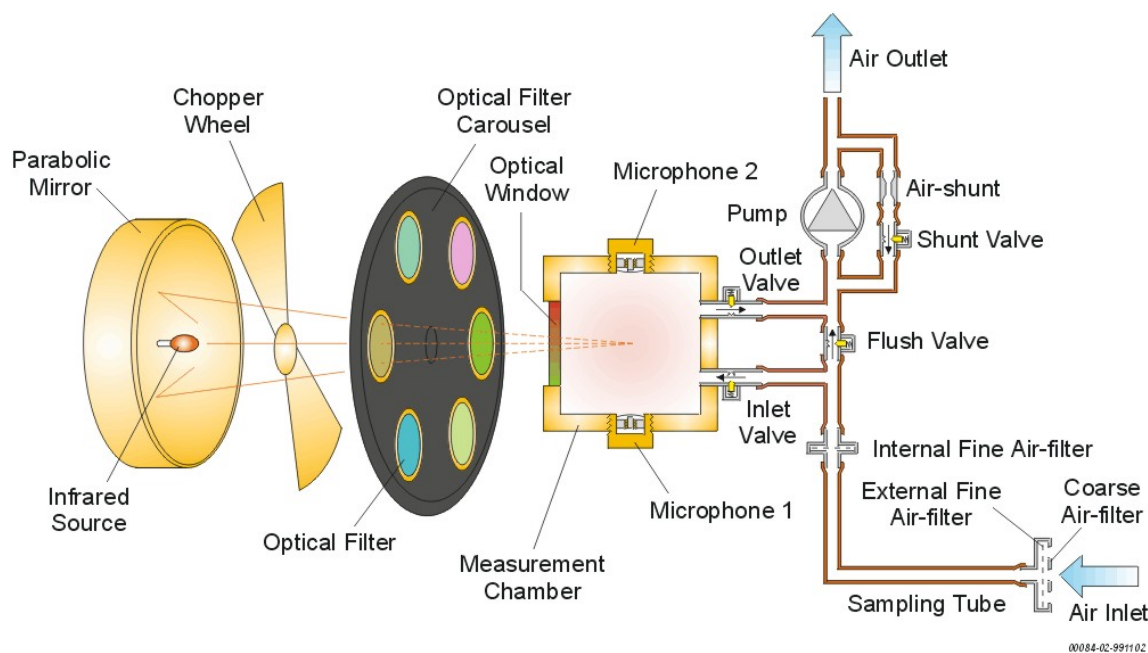


Figure 1. Schematic diagram of the major internal components of the INNOVA Model 1412 Photoacoustic Multi-gas Monitor.

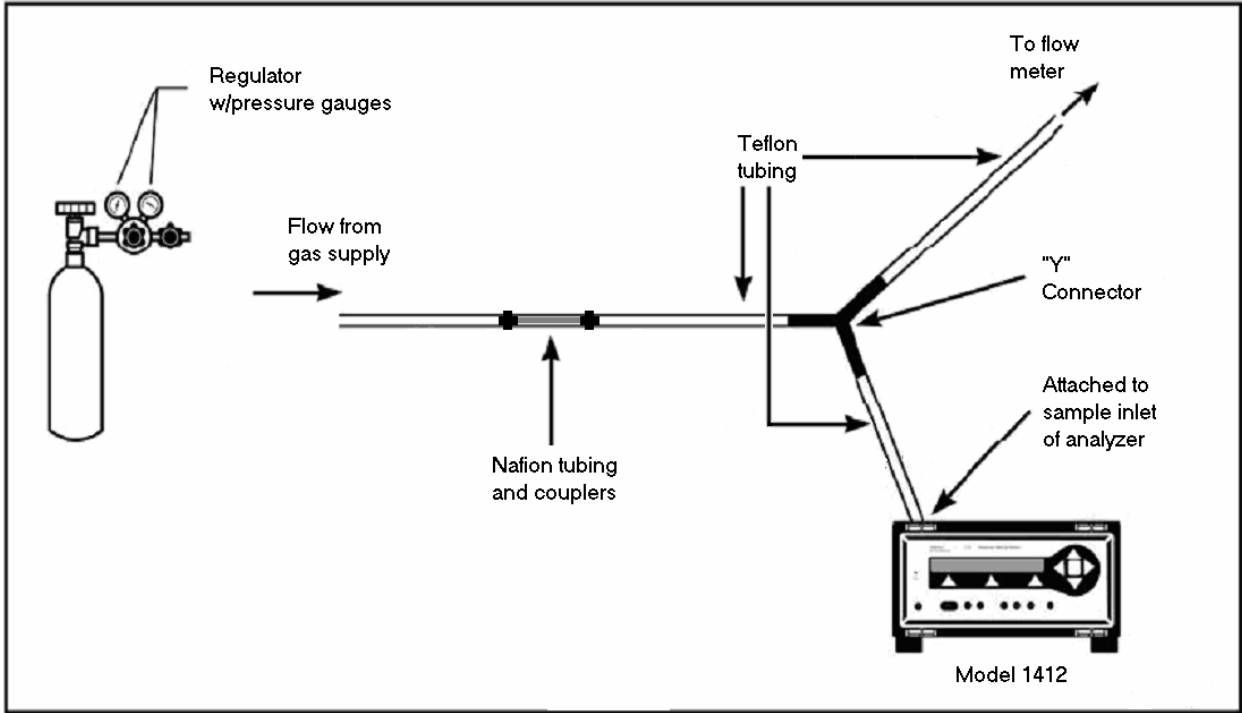


Figure 2. General equipment required for calibration of the Model 1412. Nafion tubing is coupled into the Teflon line from the cylinder during calibration of CO₂ or methane.

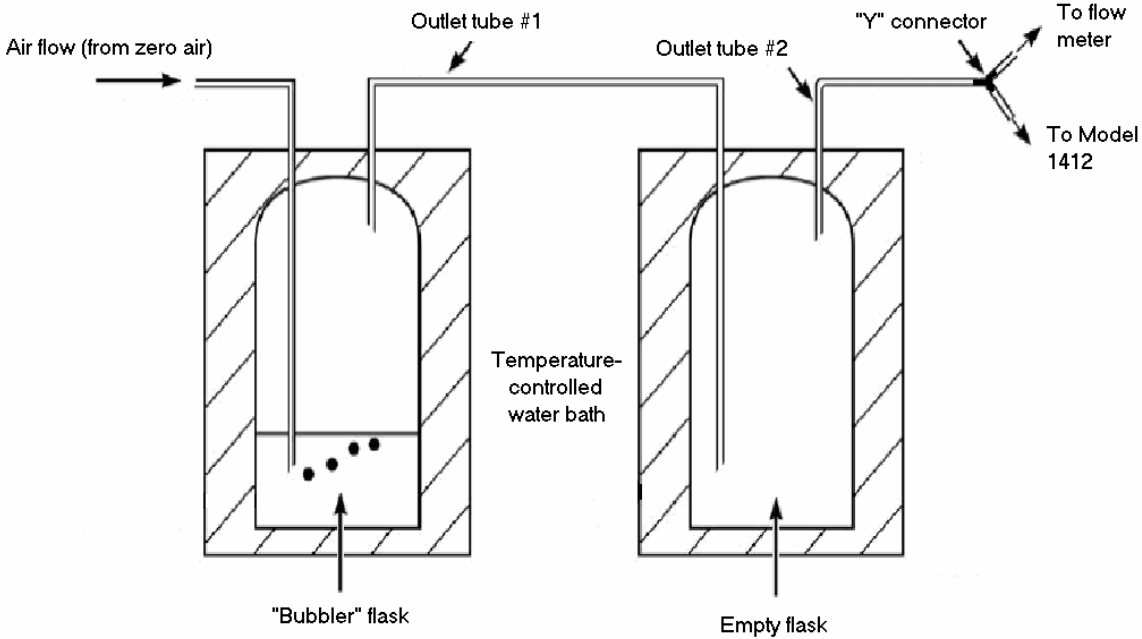


Figure 3. Schematic diagram of the equipment necessary to produce a supply of clean, wet air for humidity interference calibration.

MULTIPOINT CALIBRATION OF GAS ANALYZERS

Standard Operating Procedure (SOP) G8

MULTIPOINT CALIBRATION OF GAS ANALYZERS

Standard Operating Procedure (SOP) G8

Prepared by

Sam M. Hanni, Bill W. Bogan, and Claude A. Diehl

Reviewed by

Albert J. Heber

Effective Date: December 28, 2007

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions	4
4. Cautions	4
5. Personnel Qualifications.....	4
6. Equipment and Supplies	4
7. Procedures	5
8. Data and Records Management	7
9. Quality Control and Quality Assurance	8
10. References.....	8

1. Scope and Applicability

- 1.1. This method challenges gas analyzers with known concentrations of gases to calibrate and verifying previous calibrations of gas analyzers.
- 1.2. A multipoint calibration of gas analyzers has three objectives.
 - 1.2.1. It provides a mathematical model (generally a linear function) to convert analyzer response (signal output) to gas concentration. This model is called a calibration curve.
 - 1.2.1.1. The calibration curve is used to calculate the concentration of an unknown sample from the analyzer response.
 - 1.2.2. Linearity can be assessed, in principle, using any number of points greater than 2.
 - 1.2.2.1. The curve's goodness-of-fit indicates the linearity of the signal-to-concentration relationship.
 - 1.2.3. With replications, it provides a measure of the reproducibility of the gas analysis.
- 1.3. Accurate multipoint calibration ensures accurate gas measurements.
- 1.4. This method applies to most gas analyzers, including:
 - 1.4.1. MSA Model 3600 Photoacoustic Infrared CO₂ Monitor (SOP G3)
 - 1.4.2. TEI Model 17C Chemiluminescence (NH₃) Analyzer (SOP G4)
 - 1.4.3. TEI Model 450I Pulsed-Fluorescence (H₂S) Analyzer (SOP G5)
 - 1.4.4. TEI Model 55C Methane/Non-Methane Hydrocarbon Analyzer (SOP G6)
 - 1.4.5. INNOVA 1412 Photoacoustic Infrared Multi-Gas Monitor (SOP G7)
 - 1.4.6. MSA Chillgard Photoacoustic Infrared (NH₃) Analyzer (SOP G10)

2. Summary of Method

Calibration curves are obtained by measuring the signals obtained with a series of standards of known concentration. A programmable diluter, such as the Environics Model S-4040 (SOP G11), can be used to generate the desired gas concentrations for the calibration curve, or gases of known concentration can be used directly. Concentration and signal data are input into the Multipoint worksheet in the Instrument Performance Record file to generate a linear regression, and the slope and intercept are entered into the AirDAC program (SOP B2) that applies the calibration curve to the collected analyzer signals.

Multipoint calibration consists of measuring an analyzer's response to known concentrations of gas over a selected range of concentrations which is appropriate for the gas analyzer. The calibration range's upper limit is chosen to be greater than the estimated actual maximum measurement to be encountered in the field, and the lower limit is set to zero. A minimum of two gas concentrations of (0 and 90% of the selected range) must be used to determine the calibration curve. If the number of gas concentrations in the calibration is greater than two, the additional points should be evenly spaced between 0 and 90%. Thus, the appropriate concentrations for a four-point calibration would be 0, 30, 60, and 90% of the selected range. If, for example, the maximum concentration of ammonia which is expected to occur in field samples is 28 ppm, an appropriate maximum for the calibration range would be 30 ppm, and the gas concentrations used for calibration would then be 0, 9, 18, and 27 ppm. The multipoint calibration requires that the series of known concentrations be measured twice.

3. Definitions

- 3.1. Range The upper and lower limits of the concentrations which a gas analyzer can accurately quantitate
- 3.2. AirDAC Air Data Acquisition and Control computer program (SOP B2)
- 3.3. GSS Gas Sampling System (SOP G1)
- 3.4. PAAQL Purdue Agricultural Air Quality Laboratory
- 3.5. QAPP Quality Assurance Project Plan
- 3.6. Zero-precision A two-point check including zero air and one known non-zero concentration of reference gas

4. Cautions

- 4.1. Lack of pressure from the gas supply will cause an inaccurate reading.
 - 4.1.1. Make sure cylinder(s) and regulator(s) are open (SOP G2).
 - 4.1.2. Check for leakage caused by improperly connected tubing.
 - 4.1.3. Check for blockage or kinks in the tubing.
 - 4.1.4. If the flow of reference gas is too low, backflow from the exhaust to the instrument can occur.
- 4.2. If a diluter is being used, ensure that the correct reference gas concentration is entered into the diluter software (SOP G11).
- 4.3. Do not use any gas cylinder below 200 psi, because the certification of the reference gas is not valid below 200 psi.

5. Personnel Qualifications

- 5.1. Personnel must be trained in the use of each individual gas analyzer before working with it. This includes reading and understanding the manual and the relevant SOP.
- 5.2. Each analyst must also read and understand this SOP before conducting a multipoint calibration.

6. Equipment and Supplies

- 6.1. Cylinder(s) containing reference gas(es) with appropriate regulator(s) (SOP G2).
- 6.2. Diluter if gas dilution is required (Model S-4040, Environics, Tolland, CT) (SOP G11)
- 6.3. Computer and AirDAQ program (SOP B2).
- 6.4. Teflon tubing
- 6.5. Fittings
- 6.6. Gas analyzers: Any subset of those listed in Section 1.4.

7. Procedures

- 7.1. Make connections for introducing the reference gas to the analyzer.
 - 7.1.1. Refer to SOP G1 for descriptions of the various options for introducing a reference gas to the analyzer, and to the QAPP for the required gas introduction point. Unless the QAPP for the particular project specifies that a different point is used to introduce the gas, introduce the reference gas at the sampling inlet.
 - 7.1.2. If a diluter is used, connect the reference gas and zero air cylinders to the diluter, and the diluter to the gas introduction point (SOP G11).
 - 7.1.3. If no diluter, connect the cylinder directly to the gas introduction point.
- 7.2. Record all times, observations, and tasks during a multipoint calibration.
- 7.3. Open the valves of the gas cylinders. Use only certified gases.
- 7.4. Open the AirDAC program (SOP B2).
 - 7.4.1. Go to the DAQ tab, and change the slope to 1 and the intercept to 0 for the analyzer that is being calibrated. Press *Apply and Save* on the DAQ tab.
 - 7.4.2. Press the “Auto Smpl” button in the upper right hand corner of the page to switch to manual sampling.
 - 7.4.3. Select “Gas Cal” (Gas Calibration) from the drop-down window next to the “Manual smpl” button. This turns on the correct solenoids in the Gas Sampling System (GSS) for the calibration.
 - 7.4.4. Record in the field notes the time that Gas Cal is started.
- 7.5. Challenge the gas analyzer with reference gases.
 - 7.5.1. If a diluter is being used:
 - 7.5.1.1. The minimum number of calibration points to be performed is three (3). The concentrations will include zero concentration, approximately 90% of selected range of the analyzer, and one or more concentrations relatively evenly spaced between the zero and 90% concentrations. The flow rate of each reference gas must be at least 0.5 L/min more than the sum of flow rates of all gas analyzers drawing from the reference gas supply (when GSS is not used), or at least 0.5 L/min more than the GSS sample flow rate when using the GSS to draw from the reference gas supply.
 - 7.5.1.2. Analyze the different concentrations sequentially in increasing order (from zero to 90%), then repeat the sequence once more to determine repeatability.
 - 7.5.1.3. Open the Environics window (SOP G11) and select the Run menu.
 - 7.5.1.4. Select Program from the Run menu.
 - 7.5.1.5. Open up the drop-down window under “Name”
 - 7.5.1.5.1. Click the title with the desired gas and Regression (e.g., “NH₃ Regression”).
 - 7.5.1.5.2. Press the “Start” button to begin gas flow (Each reference gas concentration is supplied to the analyzer for a specified duration, which is set in the Environics diluter software (SOP G11). Start with CEM Zero gas.
 - 7.5.1.5.3. Record the following information into a table which is located in the field notes on the worksheet titled Calibration (Table 1).
 - 7.5.1.5.3.1. Date
 - 7.5.1.5.3.2. The start and end times of the reference gas concentrations supplied to the analyzers (Use a date/time format)

- 7.5.1.5.3.3. Analyzer
- 7.5.1.5.3.4. Reference gas (i.e. NH₃, H₂S, CO₂, etc.) used
- 7.5.1.5.3.5. Applied reference gas concentration
- 7.5.1.5.3.6. Analyzer concentration reading at the end time for each reference gas
- 7.5.1.5.3.7. AirDAC concentration reading at the end time for each reference gas
- 7.5.1.5.3.8. AirDAC signal reading at the end time for each reference gas
- 7.5.1.6. The program will automatically stop when it has completed the calibration.
- 7.5.2. If no diluter is used, conduct a manual multipoint gas analyzer calibration as follows:
 - 7.5.2.1. The minimum number of calibration points to be performed is two (2). The concentrations will include zero and span concentrations of the reference gases.
 - 7.5.2.2. Analyze each concentration sequentially, from zero to span. Repeat once
 - 7.5.2.3. Connect the CEM Zero cylinder to the gas introduction point (SOP G1).
 - 7.5.2.4. Open CEM Zero gas cylinder for the appropriate sampling period at a flow rate 0.5 L/min in excess of the total (combined) flow rate required by all the gas analyzers drawing from the calibration line (if no GSS is being used), or 0.5 L/min in excess of the GSS flow rate (if a GSS is in use).
 - 7.5.2.4.1. Record the following information into a table, titled “Calibration”, which is located in the field notes (Table 1).
 - 7.5.2.4.1.1. Date
 - 7.5.2.4.1.2. The start and end times of the reference gas concentration supplied to the analyzers (Use a date/time format).
 - 7.5.2.4.1.3. Analyzer
 - 7.5.2.4.1.4. Reference gas (i.e. zero NH₃, H₂S, CO₂, etc.) used
 - 7.5.2.4.1.5. Applied reference gas concentration
 - 7.5.2.4.1.6. Analyzer concentration reading at the end time for each reference gas
 - 7.5.2.4.1.7. AirDAC concentration reading at the end time for each reference gas
 - 7.5.2.4.1.8. AirDAC signal reading at the end time for each reference gas
 - 7.5.2.5. After 10 min, close the CEM Zero gas cylinder and switch the connection to the span reference gas cylinder.
 - 7.5.2.6. Open the span reference gas for 10 min at the same flow rate as CEM Zero gas.
 - 7.5.2.6.1. Record the information in 7.5.2.2.1 into the “Calibration” table (Table 1).
 - 7.5.2.7. Close the span reference gas cylinder after 10 min.
 - 7.5.2.8. Repeat Steps 7.5.2.1 – 7.4.2.5 once.
 - 7.5.3. Return to the AirDAC window (SOP B2).
 - 7.5.4. Press the *Manual smpl* button in the upper right hand corner to switch back to Auto Sample mode.
 - 7.5.5. Record in the field notes the time that auto sampling is restarted.
- 7.6. Perform regression of the multipoint calibration data to obtain calibration curve.
 - 7.6.1. Go to Windows Explorer and select My Computer. Select C drive
 - 7.6.2. Select the appropriate “Project” folder (Named according to the format Project, State, Site Number; for example “NAEMS IASite4B”).
 - 7.6.3. Select the “Calibration” folder.
 - 7.6.4. Open the Instrument Performance Record File (Named according to the format Project, State, Sitenumber, Reference Gas, Instrument, Serial Number, Date (Year-Month-Day); for example “NAEMS IASite4B NH3 TE17C 305 2007-06-02.xls”).

- 7.6.5. Select the “Data” folder.
- 7.6.6. Paste a copy of the file containing the 15-s data, with the title of the project and the current date (for example “SEs20060320a.txt”), within the “Data” folder.
- 7.6.7. Open with Microsoft Excel.
- 7.6.8. Find the time that each gas concentration was supplied to the instrument. Each gas concentration is supplied for a set amount of time that is set in the Environics dilutor (SOP G11) or the QAPP.
- 7.6.9. Using the Calibration table, locate the start and end times in the data file for each reference gas that was supplied to the analyzers.
 - 7.6.9.1. Average the signal data for the last two minutes of the sampling period. The sampling period is the time that AirDAC samples a location (10 min). During the calibration, supply each reference gas for this same amount of time (10 min).
 - 7.6.9.2. Close the data file (without saving it).
 - 7.6.9.3. Enter the following data into the Multipoint Calibration worksheet (Fig. 1) of the Instrument Performance Record file for each reference gas and analyzer:
 - 7.6.9.3.1. Start and end times of each reference gas.
 - 7.6.9.3.2. Reference gas concentrations (Y*)
 - 7.6.9.3.3. AirDAC averaged signal data for each reference gas from the data file
 - 7.6.9.3.4. Analyst
 - 7.6.9.3.5. Any additional operating parameters (pressure, flow, etc.) required by each individual analyzer’s SOP
 - 7.6.9.4. The regression of the multipoint calibration data will be automatically updated.
 - 7.6.9.5. Save the slope, y-intercept, and date to the table in “Cal Record” worksheet.
 - 7.6.9.6. Save the table and chart in the “Multipoint Calibration” worksheet to the “Old Calibration” worksheet.
 - 7.6.9.7. Save the chart of the multipoint calibration in the “Graphs” worksheet.
- 7.7. Update the slope and intercept in AirDAC window (SOP B2).
 - 7.7.1. Open the AirDAC window and go to the DAQ tab.
 - 7.7.2. Enter the slope and intercept in rows 8 and 9 of the column in which the calibration regression was completed.
 - 7.7.3. Press *Apply and Save* on the DAQ tab.
 - 7.7.4. Record the slope, y-intercept, and time when settings were updated in the field notes.
 - 7.7.5. Record and save the time when the slope and y-intercept is updated in AirDAC in the “Multipoint Calibration” worksheet for the Instrument Performance Record file.

8. Data and Records Management

- 8.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
- 8.2. Manage all data according to SOP B5.
- 8.3. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes. Initial and date all corrections.

9. Quality Control and Quality Assurance

- 9.1. Conduct a multipoint calibration at the following times:
 - 9.1.1. Whenever the analyzer Control Chart (SOP Q1) indicates that a zero or precision check (SOP G9) has drifted outside the calibration limits
 - 9.1.2. After any repair of the gas analyzer
 - 9.1.3. Any time the gas analyzer has been shut down for more than 3 d
 - 9.1.4. After adjusting the sensitivity of a gas analyzer
 - 9.1.5. Any other time specified by the QAPP
- 9.2. Report QC data using a Control Chart in an Excel spreadsheet (SOP Q1).
- 9.3. Unless otherwise stated by the QAPP for the particular project, the acceptability criterion for the linear regression for a multi-point calibration (Section 7.6) is an R^2 value of 0.98.

10. References

- 10.1. SOP B2. 2006. Data Acquisition and Control Software (AirDAC). Standard Operating Procedure B2. Purdue Ag Air Quality Lab.
- 10.2. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 10.3. SOP G1. 2006. The PAAQL Gas Sampling System. Standard Operating Procedure G1. Purdue Ag Air Quality Lab.
- 10.4. SOP G3. 2006. Measurement of Carbon Dioxide (CO₂) with the MSA Model 3600 Infrared Gas Monitor. Standard Operating Procedure G3. Purdue Ag Air Quality Lab.
- 10.5. SOP G4. 2006. Measurement of Ammonia (NH₃) Using the TEI Model 17C Chemiluminescence Analyzer. Standard Operating Procedure G4. Purdue Ag Air Quality Lab.
- 10.6. SOP G5. 2006. Measurement of Hydrogen Sulfide (H₂S) with the Thermo Electron Corporation Model 450I Pulsed-Fluorescence Analyzer. Standard Operating Procedure G5. Purdue Ag Air Quality Lab.
- 10.7. SOP G6. 2006. Measurement of Methane and Non-Methane Hydrocarbons with the Thermo Electron Corporation Model 55C Analyzer. Standard Operating Procedure G6. Purdue Ag Air Quality Lab.
- 10.8. SOP G7. 2006. Use of the INNOVA 1412 Photoacoustic Multi-Gas Monitor. Standard Operating Procedure G7. Purdue Ag Air Quality Lab.
- 10.9. SOP G9. 2006. Zero and Precision Checks of Gas Analyzers. Standard Operating Procedure G9. Purdue Ag Air Quality Lab.
- 10.10. SOP G10. 2006. Measurement of Ammonia Using the MSA Photoacoustic Infrared Analyzer. Standard Operating Procedure G10. Purdue Ag Air Quality Lab.
- 10.11. SOP G11. 2006. Operation of the Environics[®] Computerized Gas Dilution System. Standard Operating Procedure G11. Purdue Ag Air Quality Lab.
- 10.12. SOP Q1. 2006. Use of Control Charts for Performance Monitoring of Gas Analyzers and Analytical Instruments. Standard Operating Procedure Q1. Purdue Ag Air Quality Lab.
- 10.13. USEPA. "Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II: Part 1." EPA-454/R-98-004. Section 12. 12.2 Multi-point Calibrations.3.

Table 1: The Multipoint Calibration Table used in the field notes.

TEI 17C Ammonia Analyzer Multipoint Calibration							
Date	10/6/2004	Analyst	Diehl	Applied	Instrument	AirDAC	
Time		Reference	Concentration	Reading	Corrected	Signal	
Started	End	Instrument	Gas	ppm	ppm/ppb	ppm /ppb	vdc or mA
10/6/2004 15:44	10/6/2004 15:54	TEI 17C NH ₃ Analyzer	CEM Zero	0	0.018	0.204	0.015
10/6/2004 15:54	10/6/2004 16:04	TEI 17C NH ₃ Analyzer	NH ₃	10	9.83	9.868	3.272
10/6/2004 17:45	10/6/2004 17:55	TEI 17C NH ₃ Analyzer	CEM Zero	0	0.004	0.185	0.008
10/6/2004 17:55	10/6/2004 18:05	TEI 17C NH ₃ Analyzer	NH ₃	9	8.77	8.843	2.925
10/6/2004 18:05	10/6/2004 18:15	TEI 17C NH ₃ Analyzer	NH ₃	16	15.97	14.608	5.207
10/6/2004 18:15	10/6/2004 18:25	TEI 17C NH ₃ Analyzer	NH ₃	24	25.2	24.990	8.369
10/6/2004 18:25	10/6/2004 18:35	TEI 17C NH ₃ Analyzer	CEM Zero	0	0.54	0.674	0.173
10/6/2004 18:35	10/6/2004 18:45	TEI 17C NH ₃ Analyzer	NH ₃	9	9.15	9.191	3.045
10/6/2004 18:45	10/6/2004 18:55	TEI 17C NH ₃ Analyzer	NH ₃	16	16.67	16.664	5.563
10/6/2004 18:55	10/6/2004 19:05	TEI 17C NH ₃ Analyzer	NH ₃	24	25.4	25.265	8.461

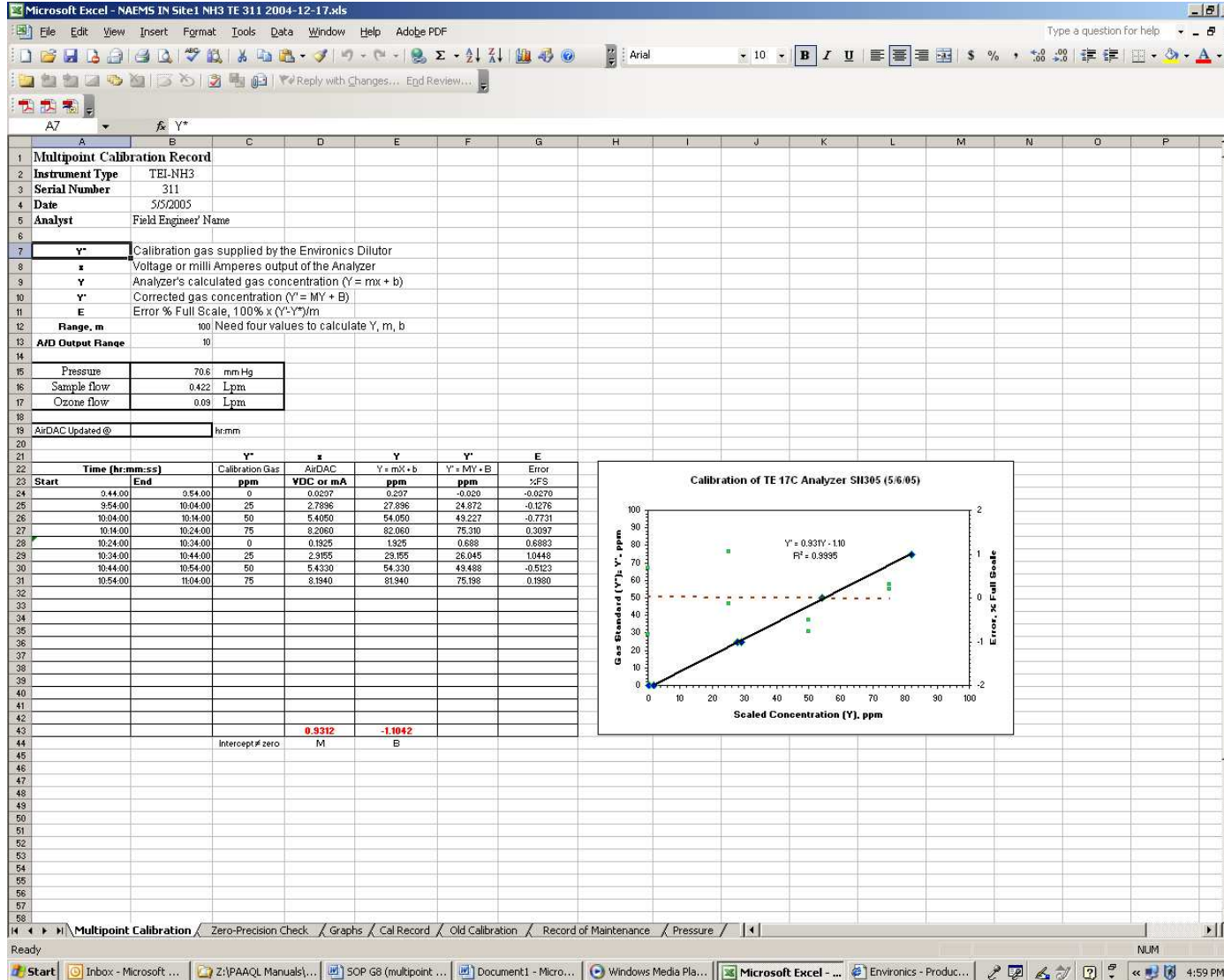


Figure 1: The Multipoint Calibration worksheet in the Instrument Performance Record File.

PRECISION CHECKS OF GAS ANALYZERS
Standard Operating Procedure (SOP) G9

PRECISION CHECKS OF GAS ANALYZERS

Standard Operating Procedure (SOP) G9

Prepared by

Bill W. Bogan and Sam M. Hanni

Reviewed by

Claude A. Diehl and Albert J. Heber

Effective Date: November 6, 2006

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions	3
4. Cautions	4
5. Personnel Qualifications.....	4
6. Equipment and Supplies	4
7. Procedures	4
8. Data and Records Management	7
9. Quality Control and Quality Assurance	7
10. References.....	8

1. Scope and Applicability

- 1.1. This method challenges a gas analyzer with known concentrations of gases to check the stability of the gas analyzer's current (most recent) full calibration.
- 1.2. Frequent precision checks of the various gas analyzers are required to determine if the calibration curves (SOP G8) used in measuring the gases of interest are still valid. This helps guarantee accuracy and quality of data generated by the analyzers.
 - 1.2.1. Zero checks indicate when an analyzer's zero reading has drifted outside of the acceptable range (which is generally approximately $\pm 2\%$ of full scale, depending on the analyzer), at which point the analyzer needs to be recalibrated.
 - 1.2.2. Span checks indicate when an analyzer drifts out of its calibration limit (which ranges from $\pm 5\%$ to $\pm 10\%$ of the baseline value, depending on the analyzer), at which point the analyzer needs to be recalibrated.
- 1.3. Zero - Span checks are used to calculate zero-offset and span factors that correct the measured gas concentrations back to the baseline measurement in the use of the CAPECAB data processing software (SOP B6).
- 1.4. This method applies to most gas analyzers, including:
 - 1.4.1. MSA Model 3600 Photoacoustic Infrared CO₂ Monitor (SOP G3)
 - 1.4.2. TEI Model 17C Chemiluminescence (NH₃) Analyzer (SOP G4)
 - 1.4.3. TEI Model 450C Pulsed-Fluorescence (H₂S) Analyzer (SOP G5)
 - 1.4.4. TEI Model 55C Methane/Non-Methane Hydrocarbon Analyzer (SOP G6)
 - 1.4.5. INNOVA 1412 Photoacoustic Infrared Multi-Gas Monitor (SOP G7)
 - 1.4.6. MSA Chillgard Photoacoustic Infrared (NH₃) Analyzer (SOP G10)

2. Summary of Method

A gas of known analyte concentration (span check) or zero air (zero check) is introduced to the appropriate gas analyzer. A diluter, such as the Environics Model S-4040 (SOP G11), can be used to generate gas concentrations required for span checks, or gases of known concentration can be used directly from the cylinder. The measured concentration at each check is entered into an analyzer-specific electronic file (the Instrument Performance Record File), which automatically updates the analyzer's Control Chart (SOP Q1).

3. Definitions

- | | |
|----------------|---|
| 3.1. Range | The upper and lower limits of the concentrations which a gas analyzer can accurately quantitate |
| 3.2. AirDAC | Air Data Acquisition and Control computer program (SOP B2) |
| 3.3. CAPECAB | Calculation of Air Pollutant Emissions from Confined Animal Buildings, a custom computer program (SOP B6) |
| 3.4. GSS | Gas sampling system (SOP G1) |
| 3.5. PAAQL | Purdue Agricultural Air Quality Laboratory |
| 3.6. QAPP | Quality Assurance Project Plan |
| 3.7. Zero-span | A two-point check including zero air and one known non-zero concentration of reference gas |

4. Cautions

- 4.1. Lack of pressure from the gas supply will cause an inaccurate reading.
 - 4.1.1. Make sure cylinder(s) and regulator(s) are open (SOP G2).
 - 4.1.2. Check for leakage caused by improperly connected tubing.
 - 4.1.3. Check for blockage or kinks in the tubing.
 - 4.1.4. If the flow of reference gas is too low, backflow from the exhaust to the instrument can occur.
- 4.2. If a diluter is being used, ensure that the correct reference gas concentration is entered into the diluter software (SOP G11).
- 4.3. Do not use any gas cylinder below 200 psi because the certification of the reference gas is not valid below 200 psi.

5. Personnel Qualifications

- 5.1. Personnel must be trained in the use of each individual gas analyzer before working with it. This includes reading and understanding the manual and the relevant SOP.
- 5.2. Each analyst must also read and understand this SOP.

6. Equipment and Supplies

- 6.1. Cylinder(s) containing reference gas(es) with appropriate regulator(s) (SOP G2).
- 6.2. Diluter if gas dilution is required (Model S-4040, Environics, Tolland, CT) (SOP G11)
- 6.3. Computer and AirDAQ program (SOP B2).
- 6.4. Teflon tubing
- 6.5. Fittings
- 6.6. Gas analyzers: Any subset of those listed in Section 1.4.

7. Procedures

- 7.1. If a new baseline is being established, save the old control chart and tables in the Zero – Precision Check worksheet into the Record of Zero – Span Checks worksheet.
- 7.2. Make connections for introducing the reference gas to the analyzer.
 - 7.2.1. Refer to SOP G1 for descriptions of the various options for introducing a reference gas to the analyzer. Unless indicated otherwise by the QAPP, introduce the reference gas at a sampling tube.
 - 7.2.2. If a diluter is used, connect the reference gas and zero air cylinders to the diluter, and the diluter to the gas introduction point (SOP G11).
 - 7.2.3. If no diluter, connect the cylinder directly to the gas introduction point.
- 7.3. Record all times, observations, and tasks during the zero – span check.
- 7.4. Open the valves of the gas cylinders. Use only certified gases.
- 7.5. Open the AirDAC program (SOP B2).
 - 7.5.1. Press the “*Auto Smpl*” button in the upper right hand corner of the AirDAC window to switch to manual sampling.
 - 7.5.2. Select “*Gas Cal*” (Gas Calibration) from the drop down window next to the

- Manual smpl* button. This turns on the correct solenoids in the Gas Sampling System (GSS, See SOP G1) for a zero-span check.
- 7.5.3. Record in the field notes the time that Gas Cal was started.
- 7.6. Challenge the gas analyzer with reference gases.
- 7.6.1. If a diluter is being used:
- 7.6.1.1. Open the Envirionics window (SOP G11) and select the Run menu.
 - 7.6.1.2. Select “Program” from the Run menu.
 - 7.6.1.3. Open up the drop-down window under “Name”.
 - 7.6.1.3.1. Open the program titled “Zero – Span Check”.
 - 7.6.1.3.2. Press the “Start” button to begin gas flow (Each reference gas concentration is supplied to the analyzer for a specified duration, which is set in the Envirionics diluter software, as described in SOP G11). Start with CEM Zero gas.
 - 7.6.1.3.3. While the program is running, record the following information into a table, titled” Zero – Span Check”, which is located in the field notes (Fig. 1).
 - 7.6.1.3.3.1. Date
 - 7.6.1.3.3.2. The start and end times of the reference gas concentrations supplied to the analyzers (Use a date/time format)
 - 7.6.1.3.3.3. Analyzer
 - 7.6.1.3.3.4. Reference gas (i.e. NH₃, H₂S, CO₂, etc.) used
 - 7.6.1.3.3.5. Applied reference gas concentration
 - 7.6.1.3.3.6. Analyzer concentration reading at the end time for each reference gas
 - 7.6.1.3.3.7. AirDAC concentration reading at the end time for each reference gas
 - 7.6.1.3.3.8. AirDAC signal reading at the end time for each reference gas
 - 7.6.1.4. The program will automatically stop when it has completed the check.
 - 7.6.1.5. Repeat Steps 7.4.5.3.2 through 7.4.5.4 for each analyzer.
 - 7.6.2. If a diluter is not used, conduct a manual zero - span check as follows:
 - 7.6.2.1. Connect the CEM Zero cylinder to the gas introduction point (SOP G1).
 - 7.6.2.2. Open CEM Zero gas cylinder for 10 min (or duration of the ambient gas sampling period) to a flow rate of 0.5 L/min in excess of total (combined) flow rate that is required by all of the gas analyzers that are being checked.
 - 7.6.2.2.1. Record the following information into a table, titled “Zero-Span Check”, which is located in the field notes (Fig. 1).
 - 7.6.2.2.1.1. Date
 - 7.6.2.2.1.2. The start and end times of the reference gas concentrations supplied to the analyzers (Use a date/time format)
 - 7.6.2.2.1.3. Analyzer
 - 7.6.2.2.1.4. Applied reference gas (i.e. zero gas, NH₃, H₂S, CO₂, etc.)
 - 7.6.2.2.1.5. Applied reference gas concentration
 - 7.6.2.2.1.6. Analyzer concentration reading at the end time for each reference gas
 - 7.6.2.2.1.7. AirDAC concentration reading at the end time for each reference gas
 - 7.6.2.2.1.8. AirDAC signal reading at the end time for each reference gas

- 7.6.2.3. Close the CEM Zero gas cylinder
- 7.6.2.4. Repeat steps 7.5.6.2 to 7.5.6.4 for each reference gas required by the gas analyzers using the sampling period of gas measurement.

Zero - Precision Check							
Date	3/12/2006	Analyst	Diehl	Instrument		AirDAC	
Time		Reference		Applied Concentration		Corrected Concentration	Analog Output
Started	End	Instrument	Gas	ppm /ppb	ppm /ppb	ppm /ppb	vdc / mA
3/12/2006 14:05	3/12/2006 14:18	TEI 17C	CEM Zero	0	0.233	-0.399	-0.047
3/12/2006 14:05	3/12/2006 14:18	MSA NH3	CEM Zero	0	8	7.42	0.078
3/12/2006 14:05	3/12/2006 14:18	TEI 45C (H2S)	CEM Zero	0	-3.1	2.55	0.000
3/12/2006 14:05	3/12/2006 14:18	TEI 45C (SO2)	CEM Zero	0	0	0.98	0.001
3/12/2006 14:05	3/12/2006 14:18	MSA CO2	CEM Zero	0	0	-37.82	3.939
3/12/2006 14:19	3/12/2006 14:34	TEI 17C	NH3	40.6 ppm	42.5	42.44	8.526
3/12/2006 14:19	3/12/2006 14:34	MSA NH3	NH3	40.6 ppm	50	49.58	0.004
3/12/2006 14:35	3/12/2006 14:46	TEI 45C (H2S)	H2S	2080 ppb	2090	2101.22	2.090
3/12/2006 14:35	3/12/2006 14:18	TEI 45C (SO2)	H2S	2080 ppb	10	10.78	0.011
3/12/2006 14:46	3/12/2006 14:57	TEI 45C (H2S)	SO2	1900 ppb	-106	-107.74	-0.109
3/12/2006 14:05	3/12/2006 14:18	TEI 45C (SO2)	SO2	1900 ppb	1907	1904.71	1.912
3/12/2006 14:58	3/12/2006 15:04	MSA CO2	CO2	4030 ppm	4000	4416	10.659

Figure 1. A Zero-Span Check table as it should be entered in the field notes.

- 7.6.3. Return to the AirDAC window (SOP B2).
- 7.6.4. Press the *Manual smpl* button in the upper right hand corner to switch back to Auto Sample mode.
- 7.6.5. Record in the field notes the time that auto sampling is restarted.
- 7.7. Record the data from the zero-span check(s) for each reference gas in the zero-span check worksheet in the pertinent Instrument Performance Record File.
- 7.7.1. Go to Windows Explorer and select My Computer.
- 7.7.2. Select C drive.
- 7.7.3. Select the appropriate "Project" folder (Named according to the format Project, State, Site Number; for example "NAEMS IASite4B").
- 7.7.4. Select the "Calibration" folder.
- 7.7.5. Open the appropriate Instrument Performance Record File (Named according to the format Project, State, Site, Reference Gas, Instrument, Serial Number, Date (Year-Month-Day); for example "NAEMS IASite4B NH3 TE17C 305 2007-06-02.xls").
- 7.7.6. Select the "Data" folder.
- 7.7.7. Paste a copy of the file containing the 15-s data, with the title of the project and the current date (for example "SEs20060320a.txt"), within the "Data" folder.
- 7.7.8. Open with Microsoft Excel.
- 7.7.9. Using the zero-span table (See the table in the field notes – Fig. 1), locate the start and end times in the data file for each reference gas supplied to the analyzers.
- 7.7.10. Average all valid measured concentration and signal data for each reference gas that is supplied during the zero – span check. Each reference gas is supplied to the GSS, either via a diluter or manually, for the same amount of time as AirDAC samples each location (i.e., 10 min).
- 7.7.11. Close the data file (without saving it).

- 7.7.12. Open the Instrument Performance Record file for each gas analyzer.
- 7.7.13. Enter the following data into the zero-span check worksheet (Fig. 2) in the Instrument Performance Record File for each instrument and reference gas:
 - 7.7.13.1. Start and end times of each reference gas supplied to the gas analyzers.
 - 7.7.13.2. Reference gas concentrations
 - 7.7.13.3. Analyzer concentration reading
 - 7.7.13.4. AirDAC averaged concentration for each reference gas from the data file
 - 7.7.13.5. Analyst
 - 7.7.13.6. Any other parameters required by each individual gas analyzer's SOP
- 7.8. The Control Chart (SOP Q1) for the individual gas analyzer will automatically update as this data is entered into the zero-span check worksheet.
- 7.9. Inspect the instrument's Control Chart in the zero-span check worksheet in the Instrument Performance Record File.
 - 7.9.1. If the zero-span check is within calibration limits, the calibration curve is valid.
 - 7.9.2. If the zero-span check is outside the calibration limits of the baseline measurement, the calibration curve is invalid. Recalibrate the analyzer (See SOP G8 and the SOP for the individual analyzer).
- 7.10. Repeat Steps 7.7.9 to 7.9.2 for each instrument, and each for reference gas.
- 7.11. Close the copy of the data file, and do not save the copy.
- 7.12. Save changes to the Instrument Performance Record File and close.

8. Data and Records Management

- 8.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
- 8.2. Manage all data according to SOP B5.
- 8.3. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes. Strike out all errors in writing with a single line. Initial and date all such corrections.

9. Quality Control and Quality Assurance

- 9.1. Conduct zero and span checks at the following times:
 - 9.1.1. Before a calibration adjustment of any gas analyzer
 - 9.1.2. Following a multipoint calibration
 - 9.1.3. Before and after any repair of the gas analyzer
 - 9.1.4. Before shutting down a gas analyzer
 - 9.1.5. After turning on a gas analyzer and warming it up
 - 9.1.6. Before the cylinder pressure drops below 200 psi, requiring its replacement.
 - 9.1.7. Upon the introduction of a new gas cylinder
 - 9.1.8. Before and after adjusting the sensitivity of a gas analyzer
 - 9.1.9. Any other time specified by the QAPP
- 9.2. Enter beginning and end times of all zero-span checks (both regularly scheduled and otherwise) immediately after completion, so the Control Chart is kept up-to-date.

10. References

- 10.1. SOP B2. 2006. Data Acquisition and Control Software (AirDAC). Standard Operating Procedure B2. Purdue Ag Air Quality Lab.
- 10.2. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 10.3. SOP B6. 2006. Data Processing Software (CAPECAB). Standard Operating Procedure B6. Purdue Ag Air Quality Lab.
- 10.4. SOP G1. 2006. The PAAQL Gas Sampling System. Standard Operating Procedure G1. Purdue Ag Air Quality Lab.
- 10.5. SOP G3. 2006. Measurement of Carbon Dioxide (CO₂) with the MSA Model 3600 Infrared Gas Monitor. Standard Operating Procedure G3. Purdue Ag Air Quality Lab.
- 10.6. SOP G4. 2006. Measurement of Ammonia (NH₃) Using the TEI Model 17C Chemiluminescence Analyzer. Standard Operating Procedure G4. Purdue Ag Air Quality Lab.
- 10.7. SOP G5. 2006. Measurement of Hydrogen Sulfide (H₂S) with the Thermo Electron Corporation Model 450C Pulsed-Fluorescence Analyzer. Standard Operating Procedure G5. Purdue Ag Air Quality Lab.
- 10.8. SOP G6. 2006. Measurement of Methane and Non-Methane Hydrocarbons with the Thermo Electron Corporation Model 55C Analyzer. Standard Operating Procedure G6. Purdue Ag Air Quality Lab.
- 10.9. SOP G7. 2006. Use of the INNOVA 1412 Photoacoustic Multi-Gas Monitor. Standard Operating Procedure G7. Purdue Ag Air Quality Lab.
- 10.10. SOP G8. 2006. Multi-Point Calibration of Gas Analyzers. Standard Operating Procedure G8. Purdue Ag Air Quality Lab.
- 10.11. SOP G10. 2006. Measurement of Ammonia Using the MSA Photoacoustic Infrared Analyzer. Standard Operating Procedure G10. Purdue Ag Air Quality Lab.
- 10.12. SOP G11. 2006. Operation of the Environics[®] Computerized Gas Dilution System. Standard Operating Procedure G11. Purdue Ag Air Quality Lab.
- 10.13. SOP Q1. 2006. Use of Control Charts for Performance Monitoring of Gas Analyzers and Analytical Instruments. Standard Operating Procedure Q1. Purdue Ag Air Quality Lab.

Record of Zero- Precision Checks											
Reference Gas =		NH ₃									
Zero Check					Precision Check						
CEM Zero Air (ppm)		0		Instrument Concentration Reading ppm	AirDAC Averaged Concentration ppm	Reference Gas (ppm)		40.6		Instrument Concentration Reading ppm	AirDAC Averaged Concentration ppm
Time		Start	End			Time		Start	End		
1/6/2006 21:16	1/6/2006 21:27			0.597	0.177	baseline	1/6/2006 21:27			1/6/2006 21:45	41.84
1/13/2006 16:28	1/13/2006 16:44	-0.078	-0.077		1/13/2006 16:45	1/13/2006 16:59	47.50	47.02			
1/20/2006 13:04	1/20/2006 13:22	0.287	-0.127		1/20/2006 13:23	1/20/2006 13:42	45.70	45.32			
1/30/2006 9:26	1/30/2006 9:42	0.372	-0.524		1/30/2006 9:43	1/30/2006 9:54	42.70	42.17			
2/10/2006 13:13	2/10/2006 13:29	0.277	-0.132		2/10/2006 13:29	2/10/2006 13:45	45.80	45.34			
3/1/2006 18:08	3/1/2006 18:25	0.402	-0.107		3/1/2006 18:25	3/1/2006 18:38	48.30	47.86			
3/6/2006 16:42	3/6/2006 16:52	0.445	0.023		3/6/2006 16:52	3/6/2006 17:01	49.00	48.61			
3/6/2006 17:02	3/6/2006 17:24	0.518	0.113		3/6/2006 17:24	3/6/2006 17:32	64.90	64.60			
Instrument Parameters											
Analyst		Pressure mmHg	Sample Flowrate Lpm	Ozonator Flowrate Lpm	Zero Offset	Span Factor					
Diehl		122.6	0.328	0.18	-0.18	0.99					
Diehl		116.4	0.329	0.219	0.08	0.86					
Diehl		123.7	0.329	0.181	0.13	0.89					
Diehl		142.4	0.327	0.172	0.52	0.95					
Diehl		134.7	0.323	0.208	0.13	0.89					
Diehl		142.2	0.314	0.187	0.11	0.85					
Diehl		149.5	0.259	0.172	-0.02	0.84					
Diehl		102.3	0.321	0.172	-0.11	0.63					

Figure 2. Example of the Zero-Span Worksheet in the Instrument Performance Record File.

**MEASUREMENT OF AMMONIA USING THE MSA
PHOTOACOUSTIC INFRARED ANALYZER
Standard Operating Procedure (SOP) G10**

**MEASUREMENT OF AMMONIA USING THE MSA
PHOTOACOUSTIC INFRARED ANALYZER
Standard Operating Procedure (SOP) G10**

Prepared by

Juan Carlos Ramirez and Claude A. Diehl

Reviewed by

Ji-Qin Ni, Bill W. Bogan, and Albert J. Heber

Effective Date: November 6, 2006

**PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)
Agricultural and Biological Engineering, Purdue University
West Lafayette, IN 47907**

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions of Terms and Acronyms	3
4. Health and Safety	3
5. Cautions	4
6. Interferences	4
7. Personnel Qualifications	4
8. Equipment and Supplies	5
9. Procedure	5
10. Data and Records Management	8
11. Quality Control and Quality Assurance	8
12. References	8
13. Contact Information	9

1. Scope and Applicability

- 1.1. The purpose of this procedure is to present the methodology and instrumentation used to measure automatically and sequentially concentrations of ammonia (NH_3) emissions at animal feeding operations (AFO), specifically at the barns.
- 1.2. NH_3 concentrations encountered at livestock facilities range from normal atmospheric concentrations (which can be as low as 1.3 ppb) to over 300 ppm (inside the barns).
- 1.3. The photoacoustic method is useful for measuring NH_3 in confined animal buildings because of the low interference of water vapor, its wide measurement range, and low maintenance required even when measuring barn concentrations.
- 1.4. The procedures described in this SOP apply only to the MSA Chillgard RT Photoacoustic Infrared Gas Monitor. The manufacturer's specifications are as follows:
 - 1.4.1. The minimum detection limit of this method is 3 ppm
 - 1.4.2. The short-term stability of the method is $\pm 1\%$ and its long-term stability, including linearity, is $\pm 5\%$ in 18 mos.
 - 1.4.3. The 90% response time to a step change in NH_3 concentration is 70 s.
 - 1.4.4. The analyzer is not degraded in the presence of silicon or sulfur compounds, including H_2S , which is emitted from livestock barns.
 - 1.4.5. The selectable ranges are 0 to 1000 ppm.
 - 1.4.6. The sampling flow rate is 0.75 L/min.

2. Summary of Method

Ammonia concentration in air is measured using photo-acoustic infrared detection with a commercial analyzer (Model Chillgard RT Photoacoustic Infrared Gas Monitor, Mine Safety Appliances, Pittsburgh, PA). With this method, a gas sample is drawn continuously from the gas sampling system's analyzer manifold, and introduced into an acoustic cell, where it is exposed to pulsing infrared light of a specific wavelength. If the sample contains NH_3 , it will absorb an amount of infrared light proportional to the concentration of NH_3 in the sample. When gas molecules absorb this infrared light, their temperature rises, and a pressure wave occurs inside the acoustic cell. The audible pressure pulses corresponding to the light pulses are detected by a very sensitive microphone located inside the chamber (MSA, 2001) and are proportional to the concentration of NH_3 molecules (Fig. 1).

3. Definitions of Terms and Acronyms

- | | | |
|------|--------|--|
| 3.1. | AirDAC | Air Data Acquisition and Control program |
| 3.2. | MSA | Mine Safety Appliances, Inc. |
| 3.3. | PAAQL | Purdue Agricultural Air Quality Laboratory |
| 3.4. | QAPP | Quality Assurance Project Plan |

4. Health and Safety

- 4.1. Be careful when working with electrical power connections.
- 4.2. Properly vent analyzer exhaust to avoid exposure to noxious gases in the sample.

5. Cautions

- 5.1. Do not mount the unit to any structure that is subject to vibration.
- 5.2. Do not locate the unit near an excessive heat source, and allow at least 3 in of clearance around the unit for proper cooling.
- 5.3. Ensure the radii of all tubing are wide enough to prevent kinking or bending. Otherwise, an obstruction in the flow path may occur.
- 5.4. Thoroughly inspect contents of the packing box to avoid discarding important parts.

6. Interferences

- 6.1. Compounds or parameters that interfere with the NH₃ readings are as follows:
 - 6.1.1. Temperature ±0.3% change in reading per °C temperature change above or below that at which the analyzer was last calibrated
 - 6.1.2. Humidity <1 ppm change in reading at any humidity level (Humidity effect is compensated using an internal humidity sensor).
 - 6.1.3. Other gases See Table 1.

Table 1. Typical cross-sensitivities of various interferants with NH₃ determination using the MSA Chillgard. Values given are averages, as actual sensitivities vary among analyzers.

Interferant gas	Concentration (ppm, %)	Equivalent NH ₃ concentration (ppm)
Acetone	1000	45
Isopropanol	1000	725
Methane	2.5%	5
Methyl Ethyl Ketone	100	25
Methylene Chloride	1000	12
<i>o</i> -Xylene	100	7
Pentane	0.75%	100
Propane	0.6%	50
R11	500	50
R12	100	65
R123	100	20
R134a	100	130
R22	550	15

Test conditions: Calibrated 0-1000 ppm NH₃ in N₂. Temperature: 25°C

7. Personnel Qualifications

- 7.1. Personnel must be trained in the use of the analyzer before initiating the procedure. Training requires about 3 h.
- 7.2. Read and understand the analyzer manual and this SOP before operating this analyzer.
- 7.3. Personnel should also be trained to interpret the analyzer output signal, converted concentration data, and correction of concentrations based on calibration.

8. Equipment and Supplies

- 8.1. Ammonia analyzer (Model Chillgard RT, Mine Safety Appliances, Pittsburgh, PA)
- 8.2. Rack and rack mounts
- 8.3. 3/8" OD x 1/4" ID x 5' Teflon tubing
- 8.4. 3/8" OD x 1/4" ID x 5' PVC tubing
- 8.5. NIST-traceable gravimetric formulated gas cylinders (specific composition will be defined in the project QAPP and/or the Site Monitoring Plan)
 - 8.5.1. Gas cylinder with Span gas
 - 8.5.2. Gas cylinder with zero air
- 8.6. Spare parts that should be on hand at each monitoring site include:
 - 8.6.1. Internal Filter, Single Point, MSA Part No. 711561
 - 8.6.2. Internal Filters, all Multipoint and Single-Point Ammonia, MSA Part No. 655533
 - 8.6.3. 2.0-A, 250-V Fuse, MSA Part No. 638091
 - 8.6.4. Low Flow Switch, MSA Part No. 637824

9. Procedure

- 9.1. Unpacking the system (See Section 2 of the Instruction Manual)
 - 9.1.1. Check the shipping box for damage. Report any damage to the carrier and note it on the delivery receipt or packing slip.
 - 9.1.2. Carefully remove the analyzer from the box.
 - 9.1.3. Check that there are no parts remaining in the box.
 - 9.1.4. Do not discard the shipping box.
 - 9.1.5. Do not install or operate the analyzer if damage is suspected.
- 9.2. Initial inspection
 - 9.2.1. Inspect the interior for damaged components or missing parts. Fig. 2 shows the front and right side view of the analyzer. Fig. 3 shows a bottom view with some components identified.
- 9.3. Gas analyzer setup
 - 9.3.1. Do not mount the unit onto any structure that is subject to vibration.
 - 9.3.2. Do not locate the unit near an excessive heat source.
 - 9.3.3. Allow at least 3 in. (7.5 cm) clearance around the unit for proper cooling.
 - 9.3.4. Securely mount the analyzer vertically on the instrument rack.
 - 9.3.5. Power connection
 - 9.3.5.1. Verify the analyzer voltage requirements match the available supply voltage. It accepts either 110 VAC or 220 VAC (Fig. 4).
 - 9.3.5.1.1. Wire the analyzer for 120 V with the following connections.
 - 9.3.5.1.1.1. Power wire (Black) to L1
 - 9.3.5.1.1.2. Neutral wire (White) to can.
 - 9.3.5.1.1.3. Ground wire (Green) to ground.
 - 9.3.5.2. Power wires should enter through a different opening than for signal wires.

- 9.3.6. Signal connection
- 9.3.6.1. Connect the analog signal (4-20 mA) output of the analyzer, at output terminals 2 (I+) and 3 (RTN) on terminal strip J17 (Fig. 5), to the data acquisition system.
 - 9.3.6.2. If a 0-10 VDC output is preferred, connect instead to terminals 4 (V+) and 5 (COM) on terminal strip J17 (Fig. 5).
 - 9.3.6.3. Signal wiring should exit through an opening at the bottom of the analyzer. Do not use the same opening as that used for power wiring.
- 9.3.7. Sample tubing connection
- 9.3.7.1. Remove all caps and plugs at sample line inlets and exhaust.
 - 9.3.7.2. Connect the gas sampling system's analyzer manifold to the analyzer inlet with ¼" OD Teflon tubing, and connect the analyzer outlet to the air exhaust of the on-farm instrument shelter with ¼" ID PVC or other tubing.
- 9.3.8. Power up the analyzer, and allow it to warm up for at least 60 min before checking the concentration readings.
- 9.3.9. Enter the analyzer's analog signal and measurement ranges into the AirDAC software (SOP B2) so that concentrations can be displayed, converted and recorded.
- 9.4. Gas analyzer calibration
- 9.4.1. Calibrate the Chillgard RT following SOP G8 (multipoint calibrations) and SOP G9 (precision checks).
 - 9.4.1.1. Use QAPP-specified numbers of points and gas concentrations for the multipoint calibrations and precision checks.
 - 9.4.1.2. Become familiar with the calibration screens by stepping the monitor through the whole sequence shown in Table 2.

Table 2. Calibration Sequence Screens of the Chillgard RT.

#	Press	Result
1	ANY KEY	BRINGS UP MENU
2	CAL	BRINGS UP CAL MENU
3	USER	ALLOWS ADJUSTMENTS
4	ZERO	ALLOWS ZERO CALIBRATION
5	NEXT	PROMPT: ALARMS ARE NOW OFF
6	NEXT	PROMPT: SAMPLE POINT 1 TO BE USED
7	NEXT	PROMPT: APPLY ZERO GAS
8	NEXT	PROMPT: ADJUST OR OK READING
9	OK	PROMPT: REMOVE ZERO GAS
10	SPAN	PROMPT: GO INTO SPAN CALIBRATION
11	NEXT	PROMPT: ALARMS ARE NOW OFF
12	NEXT	PROMPT: SAMPLE POINT 1 TO BE USED
13	NEXT	PROMPT: APPLY SPAN GAS
14	NEXT	PROMPT: ADJUST OR OK READING
15	OK	PROMPT: REMOVE SPAN GAS
16	NEXT	PROMPT: ALARMS ARE NOW ACTIVE
17	NEXT	RETURNS TO NORMAL OPERATION

- 9.4.1.3. Precision checks
 - 9.4.1.3.1. Precision checks documents responses to zero and one or more span gases.
 - 9.4.1.3.2. No analyzer adjustments are made or maintenance conducted during a check.
- 9.4.1.4. The calibration involves analyzer adjustment only when:
 - 9.4.1.4.1. For reference zero gas, stabilized reading is more than 1% of the full scale (current full scale range of the analyzer) different than zero.
 - 9.4.1.4.2. For reference span gas, stabilized reading is more than 10% different than the certified concentration of the reference gas.
 - 9.4.1.4.3. If analyzer is adjusted, then record the analyzer's responses to both zero and span gases in an as-found precision check (SOP G8) immediately prior to adjusting the analyzer, even before the analyzer's first use. The as-found responses are used to validate data since the previous calibration or previous precision check.
- 9.4.2. The calibration procedure for each reference gas (zero or span) is as follows:
 - 9.4.2.1. Record calibration date and time in the electronic field notes according to SOP B2.
 - 9.4.2.2. On the analyzer's front panel, follow the screen's prompts for zero or span calibration.
 - 9.4.2.3. Press "NEXT" three times. Analyzer prompts for gas (line 7 or 13 in Table 2).
 - 9.4.2.4. Introduce reference gas to the analyzer. See SOP G1 for delivery options.
 - 9.4.2.5. The following gas delivery parameters should be recorded in the fieldnotes: pressure, flow, temperature, relative humidity (SOP G1).
 - 9.4.2.6. Wait until the display values has stabilized.
 - 9.4.2.7. Record the time and concentrations shown on the Chillgard display, and by AirDAC, in the field notebook.
 - 9.4.2.8. Press "NEXT". Chillgard will prompt: ADJUST OR OK READING (Table 2).
 - 9.4.2.8.1. Press "OK" to accept reading if the analyzer does not need to be adjusted.
 - 9.4.2.8.2. If analyzer adjustment is needed (See Section 9.2.2.3), press "ADJ":
 - 9.4.2.8.2.1. Press the "↑" or "↓" buttons to adjust the reading.
 - 9.4.2.8.2.2. Press "OK" to accept reading.
 - 9.4.2.9. Stop flow of reference gas.
 - 9.4.2.10. Press "NEXT" twice to return to Normal Operation.
 - 9.4.2.11. Record calibration end time in the lab notebook.
- 9.4.3. Repeat the steps in Sections 9.4.3 for each reference gas.
- 9.4.4. Return AirDAC to automatic sampling.
- 9.5. Precision checks
 - 9.5.1. Repeat steps in Section 9.2.2, but skip adjustment (Section 9.4.3.8.2).
 - 9.5.2. Check soon after any repair or maintenance to verify solution to problem.
 - 9.5.3. Use QAPP-specified frequency, replications and reference gas concentrations.
- 9.6. Troubleshooting (see Table 5-2 of the Analyzer Manual)
 - 9.6.1. Check wiring and power connections if there is no signal or power.
 - 9.6.2. Check pump if there is no flow or displayed concentrations are too low.
 - 9.6.3. Check filter and replace if the filter is dirty.
 - 9.6.4. Check flow path integrity if span gas concentration is under-reported, or if sample flow rate warning is displayed.

- 9.6.5. Clean or replace leaking solenoids. Solenoids (Fig. 5) that cannot close properly will cause a leakage into the acoustic cell, resulting in slow response and under-reporting of the span gas concentration.
- 9.7. Data acquisition, calculations & data reduction requirements
 - 9.7.1. Monitor a 4-20 mA analog output using AirDAC.
 - 9.7.2. Report the data to three significant digits.
 - 9.7.3. Follow SOP B4 to correct gas concentrations for calibration and air density.
- 9.8. Computer software
 - 9.8.1. AirDAC (SOP B2) for data acquisition
 - 9.8.2. CAPECAB (SOP B6) for data processing

10. Data and Records Management

- 10.1. Maintain all laboratory records in an electronic field notes spreadsheet designated for this method. Supplement this electronic record with a record book designated for the method, and include copies of the electronic record.
- 10.2. Manage data according to SOP B5 “Management of Barn Data”.
- 10.3. Document all data and information (e.g., sample collection method used) in the field data sheets, and within site logbooks with permanent ink, or in electronic field notes. Overstrike all errors in writing with a single line. Initial and date all such corrections.

11. Quality Control and Quality Assurance

- 11.1. Conduct gas analyzer checks (zero gas and one or more reference span concentrations) at a frequency specified by the QAPP.
- 11.2. Report QC data using a control chart in a Microsoft Excel spreadsheet (SOP Q1).
- 11.3. Conduct a multipoint calibration whenever the precision check of the gas analyzer drifts more than 10%.
- 11.4. Conduct a multipoint calibration when the zero check reading drifts more than 1% of full scale (or 10 ppm).
- 11.5. Gas cylinder concentrations will be verified by FTIR (SOP G12), or will be checked against cylinders which have been FTIR-verified.
- 11.6. Check the response time at a frequency specified by the QAPP.
- 11.7. Measure the gas analyzer inlet flow rate at a frequency specified by the QAPP.

12. References

- 12.1. Chillgard RT Manual. 1993. Chillgard RT Refrigerant Monitor Instruction Manual. (L) Rev. 13. Mine Safety Appliances. Pittsburgh, PA. Online at http://www.msa-northamerica.com/gasdetection_literature.html. Accessed 3/28/2006
- 12.2. Roczko, A. 2001. MSA Photoacoustic Infrared Technology for Detection of Refrigerant Gases. Mine Safety Appliances. Pittsburgh, PA. Online at <http://media.msanet.com/NA/USA/PermanentInstruments/HVACMonitors/ChillgardLERefrigerantMonitor/PhotoacousticRefrigerantWhitePaper.pdf>. Accessed 11/7/05.

- 12.3. SOP B2. 2005. Standard Operating Procedure for Data Acquisition and Control Software (AirDAC). Standard Operating Procedure B2. Purdue Ag Air Quality Lab.
- 12.4. SOP B3. 2005. Standard Operating Procedure for Air Data Pre-Processing Software (ADPP). Standard Operating Procedure B3. Purdue Ag Air Quality Lab.
- 12.5. SOP B4. 2005. Standard Operating Procedure for Calculation and Reporting of Air Emissions from Barns. Standard Operating Procedure B4. Purdue Ag Air Quality Lab.
- 12.6. SOP B5. 2005. Standard Operating Procedure for Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 12.7. SOP B6. 2005. Standard Operating Procedure for Data Processing Software (CAPECAB). Standard Operating Procedure B6. Purdue Ag Air Quality Lab.
- 12.8. SOP G1. 2006. The PAAQL Gas Sampling System. Standard Operating Procedure G1. Purdue Ag Air Quality Lab.
- 12.9. SOP G2. 2005. Standard Operating Procedure for Compressed Gas Cylinders. Standard Operating Procedure G2. Purdue Ag Air Quality Lab.
- 12.10. SOP G8. 2005. Standard Operating Procedure for Multi-Point Calibration of Gas Analyzers. Standard Operating Procedure G8. Purdue Ag Air Quality Lab.
- 12.11. SOP G9. 2005. Zero and Precision Checks of Gas Analyzers. Standard Operating Procedure GP. Purdue Ag Air Quality Lab.
- 12.12. SOP Q1. 2005. Standard Operating Procedure for Use of Control Charts for Performance Monitoring of Gas Analyzers and Analytical Instruments. Standard Operating Procedure Q1. Purdue Ag Air Quality Lab.

13. Contact Information

- 13.1. To obtain replacement parts, address the order or inquiry to:
Mine Safety Appliances Company
Instrument Division
P.O. Box 427, Pittsburgh, PA 15230
Phone: 1-800-MSA-INST
- 13.2. Manufacturer Technical Support: 1-888-672-4678
<http://www.msanorthamerica.com/customerservice.html>.
- 13.3. PAAQL: odor@purdue.edu, heber@purdue.edu, <http://www.AgAirQuality.com>

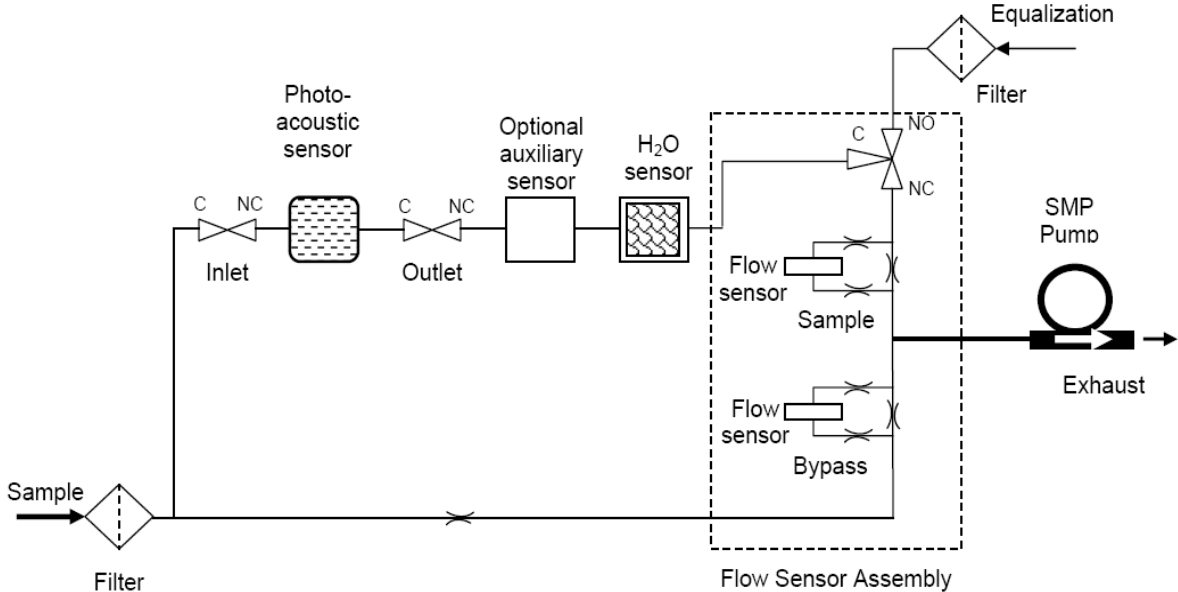


Figure 1. Analyzer flow diagram (Chillgard RT Manual, 1993).



a)



b)

Figure 2. Chillgard RT analyzer front view (a) and right side view (b).

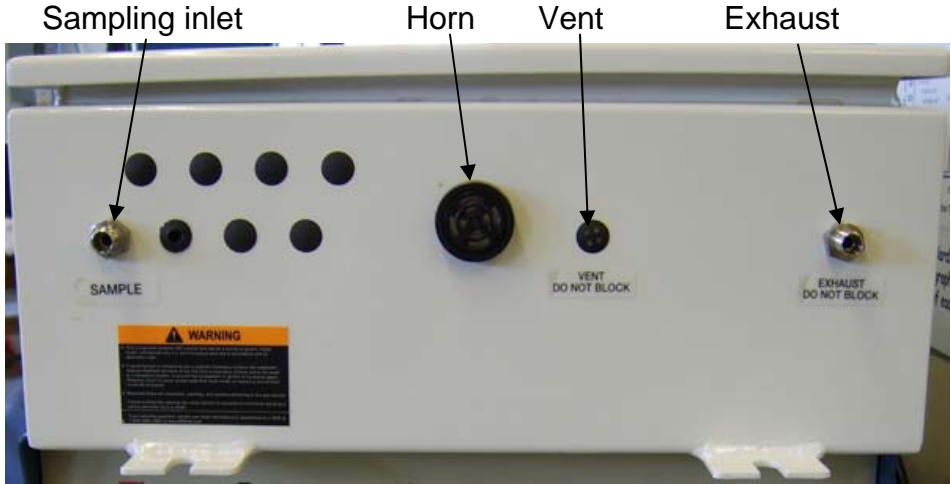


Figure 3. Chillgard RT analyzer bottom view.



Figure 4. Chillgard RT NH₃ analyzer power connections and signal outputs.

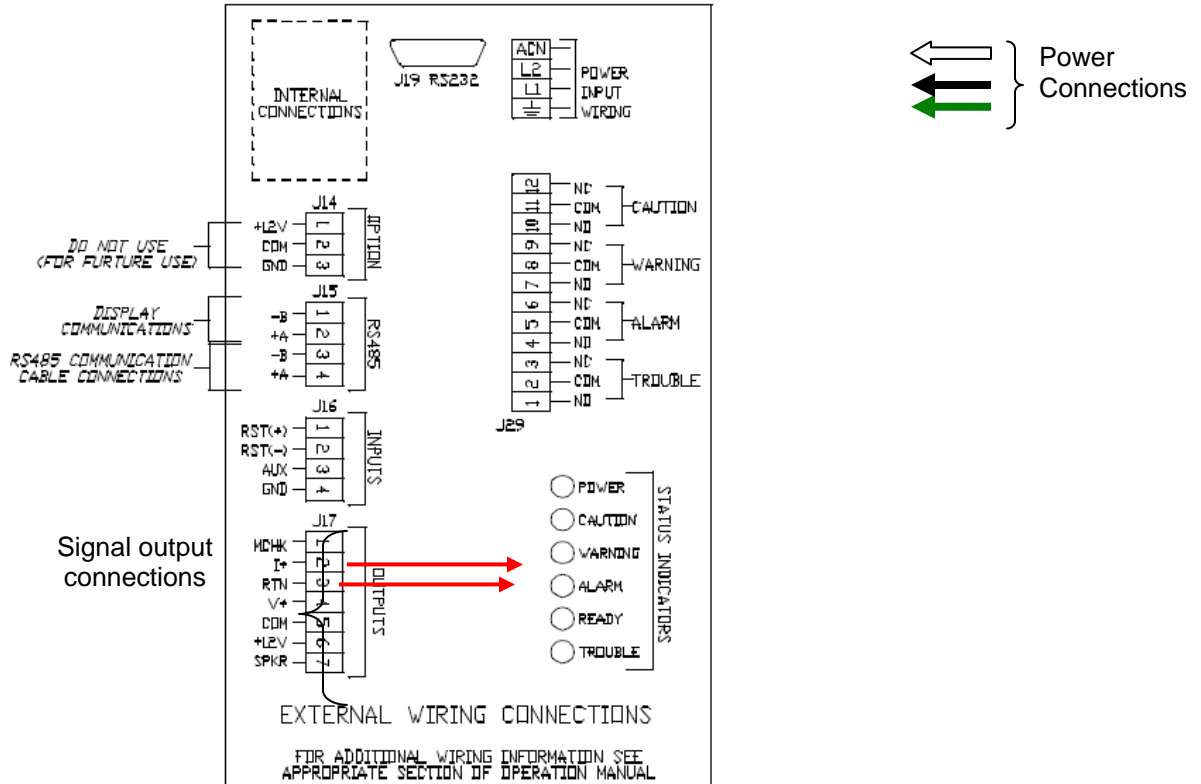


Figure 5. Wiring connections for the MSA Chillgard RT.

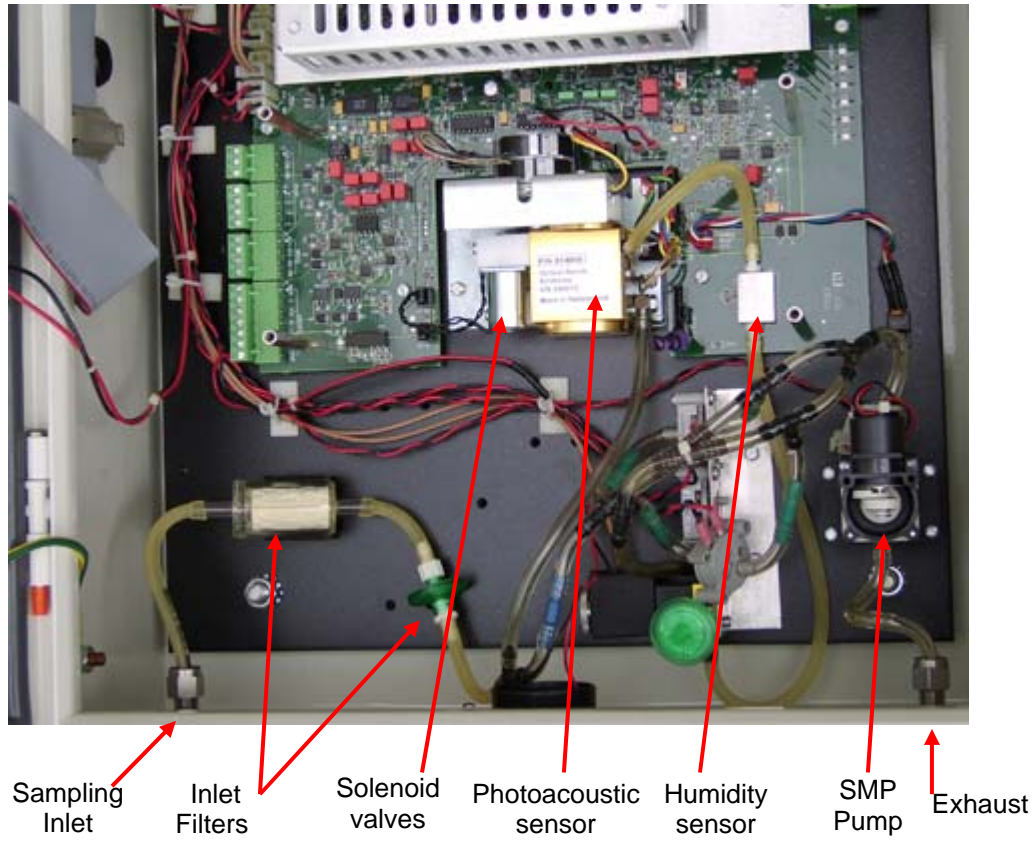


Figure 6. Chillgard RT NH₃ analyzer main components and sensors.

**OPERATION OF THE ENVIRONICS® COMPUTERIZED
GAS DILUTION SYSTEM
Standard Operating Procedure (SOP) G11**

**OPERATION OF THE ENVIRONICS[®] COMPUTERIZED
GAS DILUTION SYSTEM
Standard Operating Procedure (SOP) G11**

**Prepared by
Sam M. Hanni and Juan Carlos Ramirez**

**Reviewed by
Congna Li, Bill W. Bogan, and Albert J. Heber**

Effective Date: December 28, 2007

**PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)
Agricultural and Biological Engineering, Purdue University
West Lafayette, IN 47907**

Table of Contents

	Page
1. Scope and Applicability.....	3
2. Summary of Method.....	3
3. Definitions.....	4
4. Health and Safety.....	4
5. Cautions.....	4
6. Interferences.....	4
7. Personnel Qualifications	5
8. Equipment and Supplies	5
9. Procedure.....	6
10. Records Management	14
11. Quality Control and Quality Assurance.....	15
12. References.....	16
13. Contact Information.....	17

1. Scope and Applicability

- 1.1. This method dilutes a compressed gas standard with a balance gas, allowing the resulting mixture to be used for calibrating the continuous gas analyzers which are used in monitoring barn emissions.
- 1.2. This method applies only to the Environics Series 4040 Computerized Gas Dilution System and its accompanying computer software.
 - 1.2.1. The maximum flow rate of the gas mixture is 5 L/min.
 - 1.2.2. The gas standard is diluted to concentrations that range from 10 to 99% of its original concentration.
 - 1.2.3. The minimum concentration can be reduced to 1% of the original with the addition of one mass flow controller (MFC).
 - 1.2.4. The minimum concentration can be reduced to 0.1% of the original with the addition of two MFCs.
 - 1.2.5. The accuracy of each MFC is $\pm 1\%$ of its set flow rate.
 - 1.2.6. The accuracy of the overall dilution ratio is an additive function of the errors of all MFCs involved in the dilution. For example, if two MFCs are used, the maximal error (if the individual errors of the two MFCs are in the same direction) is $\pm 2\%$. If the errors of the individual MFCs are in the opposite directions, the overall dilution ratio error will be considerably lower than this.
 - 1.2.7. The diluter can be programmed to automatically deliver diluted standards on a schedule.
 - 1.2.8. The diluter described and used in this SOP has all its gas-accessible internal surfaces passivated with a non-reactive (RESTEK) coating, which makes it suitable for use with reactive gases.

2. Summary of Method

The Environics® Series 4040 is a computerized gas dilution system that automatically generates precise gas calibration standards for single or multi-point calibration of analyzers. The system consists of two components: the Series 4040 Diluter and the user's PC. The user interface is a Microsoft® Windows application that communicates with the Environics system via a RS232 serial interface connected to any available COM port of the personal computer. The Series 4040 instrument has a single chassis supporting up to four mass flow controllers (MFC) having ranges of 5, 5, 0.5, and 0.05 L/min, respectively. This set of flow controllers allows accurate dilution of gas standards over a very wide range of dilution ratios, by selection of the appropriate flow controllers. The MFCs are factory-calibrated using a primary flow standard traceable to the National Institute of Standards and Technology (NIST). Each flow controller utilizes an 11-point calibration table with linear interpolation, to increase accuracy and reduce flow controller nonlinearity. During operation, the protocol gas concentration, inlet port, desired output concentration, and desired output flow rate are entered by means of the keypad of the personal computer used to operate the 4040 diluter, and the diluter then sets the required standard and diluents flow rates to produce the desired mixture. The 4040 diluter indicates on the computer's display the actual concentration being produced, which in some cases differs very slightly from the nominal concentration requested. In all cases the actual

concentration produced is recorded as the concentration provided to the analyzers undergoing testing. To calibrate this instrument, it is necessary to choose a pre-calibrated gas analyzer to test the concentrations produced by the diluter.

3. Definitions

3.1.	FTIR	Fourier transform infrared spectroscopy
3.2.	GC/MS	Gas chromatography/mass spectrometry
3.3.	GSS	Gas sampling system
3.4.	K-factor	Gas correction factor
3.5.	MFC	Mass flow controller
3.6.	NIST	National Institute of Standards and Technology
3.7.	OGC	Output gas concentration
3.8.	PC	Personal computer
3.9.	QAPP	Quality Assurance Project Plan

4. Health and Safety

- 4.1. Avoid mixing gases that may produce an explosion or other hazardous reaction. Follow all other health and safety precautions related to gas cylinders (SOP G2).
- 4.2. Disconnect power before servicing the unit.
- 4.3. Make sure that replacement fuses are the correct size and rating (115 V).
- 4.4. Use only grounded outlet electrical outlets.

5. Cautions

- 5.1. Verify that scheduled calibration date is prior to the expiration date of the standard gas.
- 5.2. Verify that the instrument ambient temperature conditions are in the manufacturer's recommended range of 0°C to 50°C.
- 5.3. If the Environics software is not connected to the LabView (AirDAC) data acquisition software (SOP B2), adjust its time to agree with the time of the LabView computer.
- 5.4. Note all other cautions related to gas cylinders (SOP G2).
- 5.5. In any case where the Model 4040 is not under the control of the AirDAC program, make sure that the computer clock (used by the Environics system) is synchronized with the time in AirDAC. If the times are not synchronized, any task programmed on the Environics diluter could be out of phase with other tasks controlled by the AirDAC program.
- 5.6. Use of any USB adapter other than the one described in Section 8.3 may cause communications problems between the PC and diluter. If such problems are suspected with a different adapter, replace with this one as a first measure.

6. Interferences

- 6.1. Do not operate the diluter at a flow lower than 10% of the working range of the flow controller selected (this instrument has four flow controllers for different working

ranges), which is a flow region where flow control errors might be enhanced. The 4040 diluter provide warnings if a flow controller is being operated at this low range. Switching to another flow controller, if available, will minimize these uncertainties.

7. Personnel Qualifications

- 7.1. Each operator must read and understand this SOP, SOP G2, and the instrument manual before working with the instrument.

8. Equipment and Supplies



a)



b)

Figure 1. Front (a) and back (b) of the Environics Model 4040 Gas Diluter.

- 8.1. Environics Series 4040 Gas Dilution System
- 8.2. PC. Minimum recommended: 66-MHz 486 PC with 8 MB of RAM.
 - 8.2.1. Operating system: Windows 3.1 or newer (including Windows 95/98/Me and Windows NT/2000/XP).

- 8.3. Communications cable/adaptor: USB to Serial adapter DB9m, G-uc232a, Cyberguys.com.
- 8.4. Mass flow meter
- 8.5. Teflon tubing
- 8.6. 9/16" box wrench

9. Procedure

- 9.1. Instrument setup
 - 9.1.1. Hardware setup
 - 9.1.1.1. Connect the diluter to the computer via an RS232 cable. Connect the cable to one of the COM ports (1-4) on the computer. COM 1 is the system's default port.
 - 9.1.2. Software installation
 - 9.1.2.1. Close all running window applications.
 - 9.1.2.2. Install the Environics Series 4000 Software from the CD included with the instrument.
 - 9.1.2.3. To start the software, double-click the Environics Series 4000 icon or select Environics Series 4000 from the START menu.
 - 9.1.3. Data disk installation
 - 9.1.3.1. Instrument Data Disk Installation installs instrument specific information into the Environics Instrument Data base.
 - 9.1.3.2. Start the Environics Series 4000 Software.
 - 9.1.3.3. From the Main Menu, select Configure – Instrument – Install.
 - 9.1.3.4. Insert the Instrument Data Disk in the appropriate floppy disk drive.
 - 9.1.3.5. When the Install Instrument box appears, select the appropriate Drive Icon (A or B) from the pull down box and click OK.
 - 9.1.3.6. The instrument is now ready to use.
 - 9.1.4. Creating desktop
 - 9.1.4.1. From the Environics Series 4000 main window select File and then New.
 - 9.1.4.2. In the DESKTOP box, enter a name for the Desktop.
 - 9.1.4.3. In the INSTRUMENT box, click the drop down arrow to display the list of available instruments and select the serial number of the instrument you are using.
 - 9.1.4.4. Click OK to save the new file.
 - 9.1.5. Cylinder Configuration
 - 9.1.5.1. Open Series 4000 main window click on Configure and then Cylinder to open the cylinder library. This will bring up the Cylinder Library form (Fig. 2).
 - 9.1.5.2. Click Add and enter a name for the new cylinder at the prompt. The name of the cylinder will usually be the name of the gas of interest in the cylinder.
 - 9.1.5.3. Click the OK button. This name will appear in the Name box.
 - 9.1.5.4. K-Factor should be set to automatic.
 - 9.1.5.5. Creation Date should be the current date.
 - 9.1.5.6. If there is no expiration date on the cylinder then click on the box next to No Expiration Date. If there is an expiration date then unclick the box next to No Expiration Date and enter in the expiration date on the cylinder.
 - 9.1.5.7. Type the cylinder serial number in the Information box.

- 9.1.5.8. Enter contents of the cylinder beginning with the balance gas. Using the pull-down box under Gas Name or Symbol in the Contents section, select the desired balance gas. The concentration will be automatically set at 1 million ppm.

Interest	Balance	Gas Name	Symbol	Concentration	Units	K-Factor
<input type="checkbox"/>	<input checked="" type="checkbox"/>	NITROGEN	N2	999900.00	ppm	1.000
<input checked="" type="checkbox"/>	<input type="checkbox"/>	CARBON MONOXIDE	CO	100.00	ppm	0.9992
<input type="checkbox"/>	<input type="checkbox"/>					
<input type="checkbox"/>	<input type="checkbox"/>					
<input type="checkbox"/>	<input type="checkbox"/>					
<input type="checkbox"/>	<input type="checkbox"/>					
<input type="checkbox"/>	<input type="checkbox"/>					
<input type="checkbox"/>	<input type="checkbox"/>					
<input type="checkbox"/>	<input type="checkbox"/>					
<input type="checkbox"/>	<input type="checkbox"/>					

Figure 2. The Cylinder Library Screen.

- 9.1.5.9. Continue adding the remaining component gases by selecting them from the Gas Name or Symbol pull down box.
- 9.1.5.10. Select default gas of interest by clicking the Interest box next to the desired gas.
- 9.1.5.11. Once all gases have been added, enter the concentration of each component gas under the concentration box. The units can be changed from ppm to % using the pull down box under units. The concentration of each gas and the unit of gas will be identified on the side of the cylinder. If the gas is read in ppb then will need to be converted to ppm. The balance gas concentration will automatically be filled in as the difference so that all gases add up to 1 million ppm (100%).
- 9.1.5.12. As data is entered, the cylinder K-factor will automatically be computed based on the concentration of the individual gases in the cylinder.
- 9.1.5.13. When done, click Close to save the cylinder information and exit the Cylinder Library. The Program will display a prompt to allow the new cylinder to be saved. Click the Yes button to save.
- 9.1.5.14. Alternately, click Add to add a new cylinder without exiting the Cylinder Library.
- 9.1.6. Editing cylinder gas information
- 9.1.6.1. To move or delete a gas, click the name of the gas. The name box will turn red.

9.1.6.2. To move a gas, click on the Move Up or Move Down button to move the gas down one position on the list. Continue to click on the Move Down button until the gas is in the desired location on the list.

9.1.6.3. To delete the selected gas from the cylinder, click the Clear button.

9.1.7. Port configuration

9.1.7.1. To open the Port Configuration screen, select Configure – Port from the main menu. The desktop must be open to change the port configuration. If a desktop is not open, then Configure – Port will not be accessible.

9.1.7.2. The current cylinder assignments for the port configuration are displayed in the port configuration assignment table (Fig. 3). To assign a different cylinder to the port, uses the pull down box under cylinder, and select the desired cylinder from those listed in the cylinder library.

9.1.7.3. Once the cylinder is assigned, the default gas, symbol, concentration, and K-factor are automatically placed in the table from the cylinder library. It cannot be edited within the port configuration table. To edit this information, click on Cylinder Library and edit from within the library.

Port	Cylinder	Default Gas	Symbol	Conc	K-Factor
1	n2	NITROGEN	N2	1000000.00 ppm	1.000
2	co - 100ppm	CARBON MONOXIDE	CO	100.00 ppm	1.000
3					
4					

Figure 3. The Port Configuration Table.

9.1.7.4. To save the cylinder/port assignments, click on Close, then click YES.

9.1.7.5. Using ¼" Teflon tubing with a Gyrolok fittings provided with the instrument connect one end of the tubing to the Environics configured port and, with a Swagelok fitting connect the other end of the Teflon tubing to the regulator on the desired cylinder.

9.1.7.6. To remove cylinders from a port, select the cylinder by clicking on the cylinder name in the assignment table, and click the Clear button to remove the cylinder from the port configuration table.

9.2. Operating the instrument

9.2.1. Concentration Mode

- 9.2.1.1. To open the Concentration Mode (Fig. 4) screen, select Run – Concentration from the Series 4000 main window.
- 9.2.1.2. To add a Concentration Mode file click on Add.
- 9.2.1.3. Enter the file name and click OK.
- 9.2.1.4. A blank Concentration Mode table will appear.
- 9.2.1.5. Specify each desired source cylinder by using the pull down box in the concentration table under Cylinder. Select the cylinder by clicking on it. The port is automatically selected based on the current cylinder/port assignments. The port number is displayed in the Port box.

Balance	Cylinder	Gas	Target OGC	Units	Actual OGC	Units
<input checked="" type="checkbox"/>	n2	NITROGEN	500000.00	PPM	0.00	PPM
<input type="checkbox"/>	co - 100ppm	CARBON MONOXIDE	50.00	PPM	0.00	PPM
<input type="checkbox"/>						
<input type="checkbox"/>						
<input type="checkbox"/>						
<input type="checkbox"/>						
<input type="checkbox"/>						
<input type="checkbox"/>						
<input type="checkbox"/>						
			Total Flow	1000.00	CCM	

Port: 1 MFC: 1 Max CCM: 994.00

Run Time: 0000:00:00

Figure 4. The Concentration Mode Screen.

- 9.2.1.6. Next, specify the gas of interest by using the pull down box under Gas next to the cylinder name. Select the component gas of interest by clicking on it. To see the contents of the cylinder, click on View Cylinder.
- 9.2.1.7. Once all of the cylinders are entered, select the balance gas by clicking the balance box beside the cylinder name.
- 9.2.1.8. Specify the desired output concentration for each cylinder. Type the value in the Target Output Gas Concentration (OGC) box. If desired, the units can be changed between ppm and % by using the pull down box under units.
- 9.2.1.9. Finally, enter the total flow by typing in its value. The units can be changed from ccm to L/min by using the pull down menu under units. The value entered is used by the system to compute the target concentration of the balance gas and the required flow of each controller.

9.2.2. Program Mode

9.2.2.1. To open the Program Mode screen (Fig. 5), select Run – Program from the Series 4000 main window.

9.2.2.2. To add a Program Mode file click on Add.

9.2.2.3. Click the Mode pull down box in the row of the desired step. Select the desired run mode which will be Conc.

The screenshot shows the 'Program' window with the following data in the table:

Step	Run Time	Mode	Name	Elapsed Time
1	0000:10:00	Flow	N2 - 10 SLPM	
2	0000:01:30	Conc	N2 Zero	
3	0000:02:00	Conc	O2 Cal #1	
4	0000:05:00	Flow	N2 - 10 SLPM	
5	0000:02:00	Conc	N2 Zero	
6	0000:05:00	Conc	SO2 Cal #1	

Figure 5. The Program Mode Screen.

9.2.2.4. The available saved mode names will appear in the Name box. Open the pull down box under Name; select the desired run mode file.

9.2.2.5. Click in the Run Time box to set the run time for the mode (HH:MM:SS).

9.2.2.6. Repeat until all desired steps have been entered.

9.2.2.7. Press the Start button to begin the current program. While the program is running, the Elapsed Time box indicates the elapsed time for the current step. In addition, the Program Time box indicates the total elapsed time.

9.2.2.8. To abort the current step and proceed to the next, press the Advance button.

9.2.2.9. To stop the program before completion, click the Stop button.

9.2.3. Schedule Mode

9.2.3.1. To open the Schedule Mode screen (Fig. 6), select Run – Schedule from the Series 4000 main window.

9.2.3.2. Click Add, enter a name for the file and click OK.

9.2.3.3. A blank Schedule Mode table will appear. Fill in the table with the desired values.

Step	Runtime	Mode	Name	Start Time/Date	Set
1	0000:05:00	Flow	N2 - 10 SLPM	4:00:00 PM Daily: on every weekday	...
2	XXXXXXXXXXXX	Program	Cal prog #1	1:00:00 PM Weekly: on ,Mo,We,Fr	...
3	XXXXXXXXXXXX	Program	Cal prog #2	4:00:00 PM Bi-weekly: on ,Th	...
4	XXXXXXXXXXXX	Program	Cal prog #3	9:00:00 AM Monthly: on the 2nd Mon	...
5	0000:02:00	Conc	Q2 Cal #1	1:35:29 PM Monthly: on day 15	...
6	0000:05:00	Flow	N2 - 10 SLPM	10:00:00 AM Yearly: on day 10 of June	...
					...
					...

Figure 6. The Schedule Mode Screen.

- 9.2.3.4. Click the Mode pull down box in the row of the desired step. Select the desired run mode which will be Conc.
- 9.2.3.5. The available saved mode names will appear in the Name box. Open the pull down box under Name; select the desired run mode file.
- 9.2.3.6. Click in the Run Time box to set the run time for the mode (HH:MM:SS).
- 9.2.3.7. Click the SET button to set the time/date for this item. This will bring up the Time/Date editor (Fig. 7).
- 9.2.3.7.1. Select the type of event using the buttons on the left side of the screen. For an item that occurs only once, click the Specific Date button. For a repeating (Daily, Weekly, etc.) event, click the button for the desired frequency.
- 9.2.3.7.2. For a repeating event, fill out the appropriate options at the bottom of the screen. The options displayed will vary, depending on the type of repeat chosen. If an item is repeated weekly or biweekly, select the day(s) of the week that it is to be run by clicking in the appropriate square(s). For items repeated monthly, select the day of the month on which to repeat the item (such as every first of the month or every third Monday). For items repeated yearly, select the day and month on which to repeat the item (such as every January 1 or every third Monday in May).
- 9.2.3.7.3. Enter the desired start time, by clicking on the Start Time box and typing in the time, or by clicking the up/down arrows.

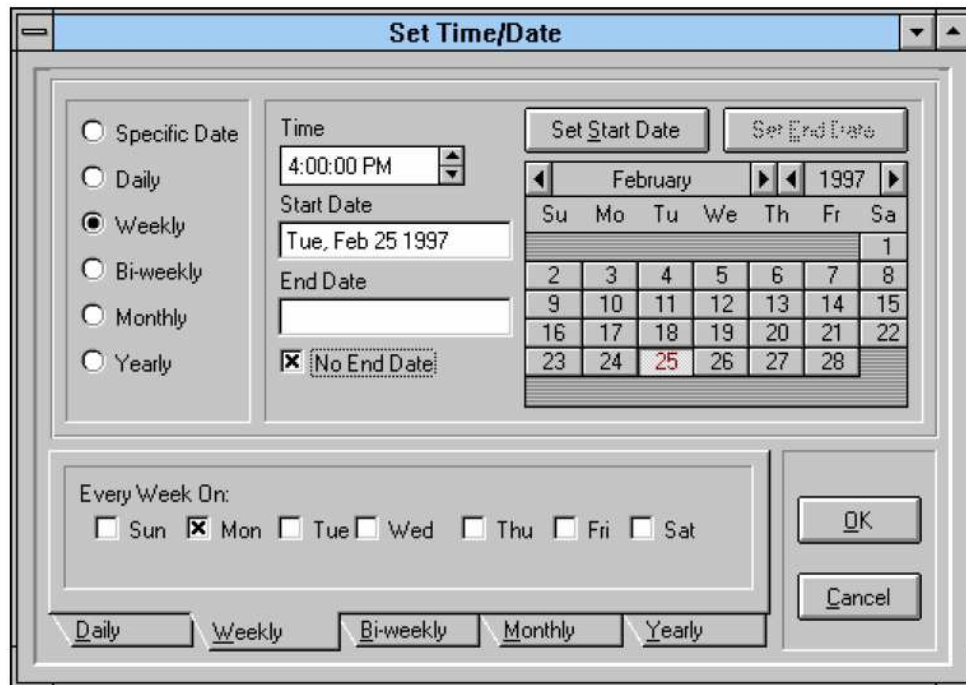


Figure 7. The Time/Date Editor within Schedule Mode.

- 9.2.3.7.4. Select the desired start date from the calendar by clicking on the day and then clicking the SET START DATE button. Use the left/right buttons to change the month or year. For a Specific Date event, this is the date the item will be run. For repeating events, it is the date the event will start to repeat.
- 9.2.3.7.5. For repeating events, select the desired end date from the calendar by clicking on the day and then clicking the SET END DATE button. This allows the repeating event to occur within a specific range of dates. Alternately, if this item is to be repeated indefinitely, click the NO END DATE box. End Date is not used for a Specific Date event.
- 9.3. 9.3. Generating reports
- 9.3.1. From the Main Menu, select Report and then the desired report type, or click the Report button directly from the Configure/Run Mode that is currently in use.
- 9.3.2. In the Reports dialog box (Fig. 8), click the Select Report dropdown box to select the desired report type. The types of available reports are given in Table 1.
- 9.3.3. Select the desired "Create Report For" and "Selection" options desired.
- 9.3.3.1. To limit the report to a specific item, click the "Current desktop, selection only" option, and select the desired item in the "Selection" box.
- 9.3.3.2. To limit the report to all items in the current desktop, click the "Current desktop, all items" option. The item shown in the "Selection" box is ignored.
- 9.3.3.3. To report on all items in all desktops, click the "All items" option. The item shown in the "Selection" box is ignored.



Figure 8. The Reports dialog box.

Table 1. Available report formats in the Environics software.

Gas Library	Lists all gases in the gas library
Cylinder Library	Shows cylinder name(s) and parameters (compositions and concentrations), for one or all cylinders
Cylinder Audit	Gives names, creation and expiration dates, and days until expiration for all cylinders
Physical Configuration	Gives configuration details for the MFCs, ports, and solenoids in the instrument
Instrument Calibration	Gives calibration tables for the MFC
Port Configuration	Gives cylinder assignments (name, gas and concentration) currently made to each port
Concentration Mode	Details one or more Concentration Mode files (cylinders, gas concentrations, total flow rate)
Programs	Details one or more Program Mode files
Schedules	Details one or more Schedule Mode files

- 9.3.4. Click Print to view the report on the screen. A sample Program Mode report is shown in Fig. 9.
- 9.3.5. Print the report and store it according to SOP B5.
- 9.3.6. Click the arrow buttons to move between pages. Click the Magnifying Glass Icon to change the view size.
- 9.3.7. Click the Printer icon to send the report to the printer.

- 9.3.8. When finished, click the Close button to return to the Reports menu.
- 9.3.9. Repeat Sections 9.6.2 through 9.6.8 to generate additional reports.
- 9.3.10. When done, click Close to return to the Main Menu.

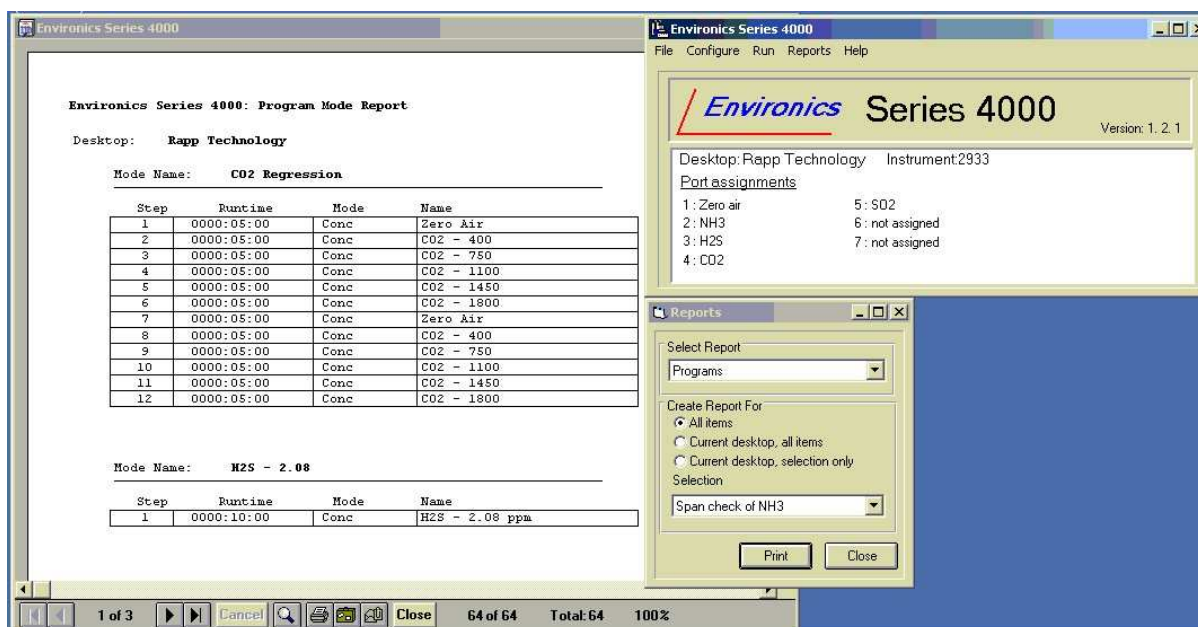


Figure 9. Screen capture showing a sample Program Mode report.

9.4. Troubleshooting

- 9.4.1. Perform the steps in Section 9.1.3 if a “no instruments registered” warning appears.
- 9.4.2. When opening a desktop, the software will try to communicate with the assigned instrument. If it is unable to communicate, a warning message will be displayed.

There are two possible reasons for this:

- 9.4.2.1. The desktop’s assigned serial number does not match the instrument being used.
- 9.4.2.2. The selected PC serial port on the EnviroNics software might be configured to a COM different from the actual COM that is connected to the PC.
- 9.4.3. The only available cylinders listed are those assigned to ports. If the desired cylinder is not listed, modify the port settings.
- 9.4.4. The Program Mode cannot be set up unless either the Concentration, Flow, or Divider mode is set up.
- 9.5. Computer hardware & software
- 9.5.1. Store all applicable software in the data acquisition computer.
- 9.5.2. The dilutor can be controlled by, and input data to, the AirDAC program (SOP B2).

10. Records Management

- 10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.

- 10.2. Manage all data according to SOP B5.
- 10.3. Document all data and information (e.g., sample collection method used) on field data sheets, and within site logbooks with permanent ink, or in electronic field notes.
- 10.4. Overstrike all errors in writing with a single line. Initial and date all such corrections.

11. Quality Control and Quality Assurance

11.1. Flow Calibration (Initial)

11.1.1. Mass flow controller zero check

- 11.1.1.1. Some MFCs require the cover to be removed to make adjustments
- 11.1.1.2. Connect the (-) lead from a voltmeter to PC402 GROUND (TP2).
- 11.1.1.3. Connect the (+) lead to each MFC Response Test Point: MFC1 (TP9), MFC2 (TP11), MFC3 (TP13), and MFC4 (TP15).
- 11.1.1.4. Verify that the voltage at each test point is $0\text{ V} \pm 0.025\text{ V}$ for a Tylan MFC and $0.01\text{ V} \pm 0.005\text{ V}$ for a Hastings MFC. If the "zero" is out of spec, adjust the MFC "zero" pot to bring it within these specified ranges.
- 11.1.1.5. After adjusting the "zero" pot, replace system cover.
- 11.1.1.6. After "zero" adjustment, wait at least one hour (1 h) for temperature to stabilize before proceeding to MFC calibration (Section 11.1.2).

11.1.2. Mass flow controller calibration

- 11.1.2.1. Connect calibration gas to the appropriate input ports for the MFCs being calibrated. The input pressure should be approximately 25 psig.
- 11.1.2.2. Connect a primary standard flow calibration device to the output of the system.
- 11.1.2.3. Be sure the instrument is connected to the PC and powered up.
- 11.1.2.4. Run the Environics Series 4000 software.
- 11.1.2.5. From the menu screen, select Configure.
- 11.1.2.6. Select Instrument.
- 11.1.2.7. Select Calibrate.
- 11.1.2.8. In the Reference Gas box, click the down arrow button and set this to match the calibration gas for that MFC.
- 11.1.2.9. In the Controller Instrument box, click the down arrow button, and select the serial number of the instrument to be calibrated.
- 11.1.2.10. In the MFC box, click the down arrow button, and select the desired Mass Flow Controller to enter data (start with MFC 1).
 - 11.1.2.10.1. If the MFC has been changed, select Init Points, enter appropriate size in Max Flow, Table will initialize.
- 11.1.2.11. Click on the 100% calibration point and press Start. Wait at least 10 min for flow to stabilize before continuing.
- 11.1.2.12. Using a mass flow meter, take a flow measurement. Measured flow value must be between 100% and 110% of the MFCs rated full-scale flow. If flow value is out of this range, adjust the MFC "span" pot.
 - 11.1.2.12.1. Some MFCs require the cover to be removed to make adjustments. In this case, after performing "span" adjustment, replace MFC cover. After adjusting "span" pot, replace system cover and wait at least 1 h for temperature to stabilize before continuing.

- 11.1.2.13. If measured flow value is acceptable, enter the measured value in the True Flow column. If not, repeat Step 9.3.11.
- 11.1.2.14. Click on the next calibration point, and press the Start button. Wait at least 2 min for flow to stabilize before continuing.
- 11.1.2.15. Take a flow measurement, and enter the value in the True Flow column.
- 11.1.2.16. Repeat Steps 9.3.14 to 9.3.15 until all calibration points have been completed.
- 11.1.2.17. Press Stop, then press Save to save the MFC calibration data. This will download the new calibration files to the instrument.
- 11.1.2.17.1. If a warning appears “CAL TABLE ERROR -3”, click OK, then save again before exiting.
- 11.1.2.18. Repeat steps 9.3.10-9.3.17 until all MFCs have been calibrated.
- 11.1.2.19. Click Close to return to the main menu screen.
- 11.2. Flow Calibration (Periodic)
- 11.2.1. The FRM 205 recommends that the gas dilution system shall be recalibrated once per calendar year using NIST-traceable primary flow standards with an uncertainty of $\leq 0.25\%$. The QAPP may specify a more frequent calibration schedule. The manufacturer will conduct this recalibration.
- 11.2.2. Keep a label (Fig. 10) affixed at all times to the gas dilution system listing the date of the most recent calibration, and the due date for the next calibration.

Calibration date: _____ Next calibration due date: _____

Figure 10. Sample calibration label.

- 11.2.3. Before sending a unit to the manufacturer for re-calibration, record the existing (as-found) flow rate (if this is not automatically recorded by AirDAC), and the resultant dilution ratio based on that flow rate.
- 11.3. At each use of the Model 4040, document its delivered flow rate to ensure that it is at least 0.5 L/min higher than the GSS flow rate (SOP G1).
- 11.4. In any case where the Model 4040 is not under the control of the AirDAC program, make sure that the computer clock (used by the Environics system) is synchronized with the time in AirDAC.

12. References

- 12.1. Environics Model 4040 Manual. 2003. Series 4000/4040 Gas Mixing System/Gas Dilution System. Environics, Inc, Tolland, CT.
- 12.2. Environics Inc. 2003. Computerized Gas Dilution System Series 4040, Online at <http://www.environics.com/4040.html>. Accessed 10 January 2006.
- 12.3. SOP B2. 2006. Data Acquisition and Control Software (AirDAC) Standard Operating Procedure B2. Purdue Ag Air Quality Lab.
- 12.4. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue

- Ag Air Quality Lab.
- 12.5. SOP G1. 2006. The PAAQL Gas Sampling System. Standard Operating Procedure G1. Purdue Ag Air Quality Lab.
 - 12.6. SOP G2. 2006. Compressed Gas Cylinders. Standard Operating Procedure G2. Purdue Ag Air Quality Lab.
 - 12.7. SOP G12. 2006. FTIR Verification of Gas Cylinder Concentration. Standard Operating Procedure G12. Purdue Ag Air Quality Lab.
 - 12.8. SOP V6. 2006. GC/MS Analysis of VOCs Collected in Sampling Canisters. Standard Operating Procedure V6. Purdue Ag Air Quality Lab.
 - 12.9. SOP V7. 2006. Cleaning, Certification and Pre-sampling Preparation of Sampling Canisters. Standard Operating Procedure V7. Purdue Ag Air Quality Lab.

13. Contact Information

- 13.1. Manufacturer:
 Environics, Inc.
 69 Industrial Park Road East
 Tolland, CT 06084-2805
 Phone: (860) 872-1111
 Fax: (860) 870-9333
 E-mail: info@environics.com
 Web Page: <http://www.environics.com>

FTIR VERIFICATION OF GAS CYLINDER CONCENTRATION
Standard Operating Procedure (SOP) G12

FTIR VERIFICATION OF GAS CYLINDER CONCENTRATION

Standard Operating Procedure (SOP) G12

Prepared by

Congna Li

Reviewed by

Bill W. Bogan, Jacek A. Koziel (Iowa State Univ.), and Albert J. Heber

Effective Date: November 6, 2006

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions of Terms and Acronyms	4
4. Health and Safety	4
5. Interferences	4
6. Cautions.....	5
7. Personnel Qualifications	6
8. Equipment and Supplies	6
9. Procedures.....	6
10. Data and Records Management	14
11. Quality Control and Quality Assurance.....	14
12. References	15
13. Contact Information.....	16

1. Scope and Applicability

- 1.1. A method is needed which will accurately analyze the concentrations of certified standard gases in cylinders purchased from outside vendors. Such a method is needed to ensure that the gas cylinders which are used for calibrations and/or performance checks of gas analyzers and other analytical instruments do, in fact, contain gases at their stated concentrations.
- 1.2. Fourier Transform Infrared (FTIR) Spectroscopy provides a reliable method for verifying gas cylinder concentrations for vapor-phase organic or inorganic compounds that absorb energy in the mid-infrared spectral region, about 800-4000 cm^{-1} . Compounds that can be checked using this method include ammonia, carbon dioxide, methane, hexane, n-butanol, acetone, ethylene, carbon monoxide, propane, and nitrous oxide.
- 1.3. Typically, the FTIR minimum detection limit is as low as 2-10 ppb. Analytical range and sensitivity depend on the frequency-dependent component absorptivity, instrument configuration, data collection parameters, and gas stream concentration.
 - 1.3.1. Instrument factors that affect performance (range, sensitivity, etc.) include:
 - 1.3.1.1. Spectral resolution
 - 1.3.1.2. Interferometer signal averaging time
 - 1.3.1.3. Detector sensitivity and response
 - 1.3.1.4. Absorption path length
- 1.4. This procedure applies only to the Nexus 670 FTIR gas spectrometer (Thermo Electron Corporation, Palatine, IL).

2. Summary of Method

The FTIR gas spectrometer (Nexus 670, Thermo Electron Corporation, Palatine, IL) allows accurate analysis of concentrations of compounds that are delivered from gas cylinders. The FTIR analyzer consists of a KBr beamsplitter, a mercury cadmium telluride High D* (MCT-High D*) detector (cooled with liquid nitrogen), an IR source, a heated stainless steel gas absorption cell, an electronic package, and a computer. The sample gas can be delivered to the gas absorption cell either directly from the gas cylinder (continuous purge mode), or from a sample bag to the vacuumed gas cell (batch mode). The latter is used if the gas cylinder is not available onsite with the FTIR. The optical bench measures the intensity of a specially encoded infrared beam after it has been passed through a sample. The resulting signal, called an "interferogram", contains information about all frequencies present in the beam. The computer reads the interferogram, uses Fourier transformation to decode the intensity information for each frequency, and presents the spectrum. Absorbance band intensities in the spectrum are related to sample concentrations by what is commonly referred to as Beer's Law. The frequency-dependent relationship between absorptivity and concentration can be determined by taking standard spectra of samples of compounds at known concentrations. Unknown compounds can be detected by matching sample absorbance bands with bands in standard spectra, and their

concentrations can be calculated by comparing sample band intensities with standard band intensities. Using OMNIC QuantPad software (Thermo Nicolet Instrument Corporation, Madison, WI), quantitative analysis methods can be created which allow analysis of spectra from gas- or condensed-phase samples using the Classical Least-Squares (CLS) quantitative analysis algorithms.

3. Definitions of Terms and Acronyms

- 3.1. FTIR Fourier Transform Infrared (FTIR) spectrometer
- 3.2. PAAQL Purdue Agricultural Air Quality Laboratory
- 3.3. Analyte A compound that the method is required to measure.

4. Health and Safety

- 4.1. When filling the detector's Dewar flask, be careful not to contact the liquid nitrogen with your skin. Wear protective gloves and goggles, and follow standard laboratory safety practices.
- 4.2. Do not touch the infrared source housing; it may be very hot. It stays hot for up to 15 min after the spectrometer is turned off.
- 4.3. Always follow the safety information that is displayed on the spectrometer itself. Do not operate a spectrometer, or any accessory, without following the safety precautions described in the manual and the documentation that came with the spectrometer.
- 4.4. Laser light exposure is possible when a beam-deflecting accessory (for example, a diffuse reflectance accessory) is installed. Never stare into the sample compartment when a beam-deflecting accessory is installed.
- 4.5. Avoid exposure to hazardous compounds when delivered from gas cylinders. Ensure that the gases are properly vented and that the gas delivery system is leak-free.

5. Interferences

- 5.1. Background interference. This results from a change in throughput relative to single beam background. In routine sampling, throughput may degrade over several hours. Periodically a new background spectrum must be collected.
- 5.2. Interference occurs when other compounds in the sample have spectral features that overlap with those of the analyte(s). Water vapor and CO₂ are common spectral interferences. The extent of interference depends on the interferent concentration, analyte concentration, and the degree of band overlap. Spectral interference can be minimized or avoided by choosing an alternate spectral region for analysis.
- 5.3. If the temperature in the gas cell is too low, condensation of the analytes and/or water vapor can occur. Set the temperature of the gas cell at 50°C to minimize condensation.

- 5.4. In addition to being a spectral interferent, condensed moisture removes soluble compounds from the gas phase by allowing them to dissolve into the separate liquid phase.
- 5.5. Ambient air can mix with the sample if the gas absorption cell valves are not leak-free when delivering the sample in batch mode from a sample bag.
 - 5.5.1. When introducing sample from a sample bag, minimize the length of inlet tubing to reduce errors in gas concentrations.
- 5.6. Samples stored in sample bags should be analyzed as soon as possible (preferably within 24-48 hrs) to avoid potential contamination of chemicals released from the sample bag itself and the loss/uptake of gases via diffusion through walls.
 - 5.6.1. If possible, controlled tests with sample bags should be performed prior to their use to collect samples, to establish that the analyte(s) of interest are not lost from the bags. See Section 11.9.

6. Cautions

- 6.1. Always ensure that the detector is cooled by liquid nitrogen before taking a spectrum.
- 6.2. The manufacturer recommends that the spectrometer power remain on, even when not in operation. Allow the spectrometer to warm up for one hour if the power has just been turned on, then align it before collecting spectra.
- 6.3. Collect a new background spectrum regularly, preferably once every 4 h during the operation of the FTIR.
- 6.4. Collect a new background spectrum if the software parameters have been changed.
- 6.5. Align the spectrometer daily for optimum performance. The spectrometer should also be aligned if the ambient temperature has changed by at least 5°C, or in such situations such as when a laser has been replaced, a beamsplitter has been installed, the spectrometer has just been moved, or the detector signal intensity has dropped significantly from its usual level.
- 6.6. Never use a flammable gas to purge a spectrometer bench. The purge gas must be free of moisture, oil and other reactive materials. Use dried air or nitrogen to purge the instrument. Other gases, even inert gases such as argon (Ar), can damage the spectrometer laser.
- 6.7. The FTIR operates reliably at ambient temperatures between 16°C and 27°C. Temperature changes may result in drift in the system response.
- 6.8. The sample spectra to be analyzed using a particular method must have the same data point spacing and the same spectral range (frequency limits) as the spectra used to calibrate the method.
- 6.9. The calibration spectra must be of pure components or pure components diluted with a solvent or gas that either doesn't absorb infrared energy or absorbs in a different region of the infrared spectrum.

7. Personnel Qualifications

- 7.1. Personnel should be trained in the use of the instrument before initiating the procedure. Training requires about three days.
- 7.2. Each analyst must read and understand the entire instrument manual and this SOP before operating it.
- 7.3. Personnel should also be trained in verifying the instrument is functioning properly, performing routine maintenance, making a calibration method for analyzing a specific component, and interpreting the analyzer output signal.

8. Equipment and Supplies

- 8.1. FTIR gas spectrometer (Model Nexus 670, Thermo Electron Corp., Palatine, IL).
- 8.2. Stainless steel gas absorption cell (Mars Model 8L/40V, Gemini Scientific Instruments, Buena Park, CA)
- 8.3. Purge gas generator (Model PCDA1112-122, MTI-Puregas, Westminster, CO).
- 8.4. Absolute pressure gauge (Automation Products Group, Logan, UT)
- 8.5. Temperature controller (Model 689-0005, Barnant Co., Barrington, IL).
- 8.6. Heat Jacket and Thermo Controller
- 8.7. Vacuum pump
- 8.8. Nitrogen gas
- 8.9. Liquid nitrogen, Dewar flask, and funnel
- 8.10. Teflon tubing
- 8.11. Calibration standard gas
- 8.12. Sample bags (if gas cylinder is offsite)
- 8.13. Diluter

9. Procedures

- 9.1. Take triplicate bag (#1, 2, and 3) samples from a gas cylinder, if the gas cylinder is offsite.
 - 9.1.1. Flush each bag thoroughly at least 3 times with ultra-high purity (UHP) grade nitrogen and vacuum completely before use. Bag volume should be ≥ 40 L.
 - 9.1.2. Turn on the gas cylinder following SOP G2.
 - 9.1.3. Connect Teflon tubing that is connected to the gas cylinder regulator to the hose connection of bag (#1) fitting (stem protruding from the side of the bag fitting).
 - 9.1.4. Open the shut-off valve of the bag (#1) by holding the side stem and turning the entire upper portion of the fitting counterclockwise one revolution. Gas from cylinder begins to fill the bag. Fill the bag to no more than 80% of its maximum volume.
 - 9.1.5. Turn the gas cylinder off and close the shut-off valve of the sampling bag (#1) by holding the side stem and turning the entire upper portion of the fitting clockwise until it is snug.
 - 9.1.6. Detach the Teflon tubing from the sample bag (#1).

- 9.1.7. Finish sampling for bags #2, and 3 by repeating the Sections 9.1.1 to 9.1.6.
- 9.1.8. Pack the filled sample bags in a rigid carton to prevent damage or discharge during shipping. Ship the samples to the laboratory by overnight delivery, but do not ship sample bags by air unless the cargo cabin of the aircraft is pressurized. Time shipping dates so that samples are received by the laboratory when someone is there to accept them.
- 9.2. Turning on the system components
 - 9.2.1. Connect external power supply.
 - 9.2.2. Turn on accessories such as the temperature controller.
 - 9.2.3. Turn on the spectrometer by pressing the power switch (I/O).
 - 9.2.4. Turn on the printer (if available).
 - 9.2.5. Turn on the computer.
 - 9.2.6. Allow at least 1 h after turn on of the spectrometer and wait until the temperature inside the gas cell stabilized at 50°C before collecting spectrum.
 - 9.2.7. Keep spectrometer on at all times for stable operation.
 - 9.2.8. If the system components are to be turned off, do so in the following order:
 - 9.2.8.1. Turn off computer.
 - 9.2.8.2. Turn off printer.
 - 9.2.8.3. Turn off spectrometer.
 - 9.2.8.4. Turn off accessories.
- 9.3. Checking the purge gas generator
 - 9.3.1. Set the pressure regulator of the purge gas generator between 20 and 40 psi.
 - 9.3.2. Set the flow meter to deliver around 30 scfh.
 - 9.3.3. Use only nitrogen or compressed air to purge the system.
- 9.4. Starting OMNIC software
 - 9.4.1. Double-click the OMNIC shortcut on the windows desktop.
- 9.5. Cooling the detector with liquid nitrogen
 - 9.5.1. Before running any samples, check whether liquid nitrogen needs to be added.
 - 9.5.1.1. Open OMNIC.
 - 9.5.1.2. Select “Experiment Setup” from the “Collect” Menu.
 - 9.5.1.3. Go to the tab labeled “Diagnostic”.
 - 9.5.1.4. If the detector signal is a flat line, liquid nitrogen must be added to the system before spectra are collected.
 - 9.5.1.5. Wait 15 min, or until the signal intensity is satisfactory before collection (the magnitude of signal changes as a function of resolution being used). A minimum signal intensity of 30% of maximum voltage is required if resolution is set at 1.
 - 9.5.1.6. Liquid nitrogen will evaporate over time, even if the FTIR does not scan. As a rule of thumb, it should be replenished after 8-10 h, even if the system has been idle during that time.
 - 9.5.2. Add liquid nitrogen
 - 9.5.2.1. ALWAYS wear protective eye wear and gloves.
 - 9.5.2.2. Fill the small liquid nitrogen Dewar cylinder 2/3 full from a large Dewar.
 - 9.5.2.3. Open the lid on the left hand side of FTIR, and pour liquid nitrogen from the small Dewar into the top hole using the special funnel provided.

- 9.5.2.4. Close the lid and return gloves/goggles to the drawer.
- 9.6. Quant Pad set up prior to collecting spectrum
 - 9.6.1. Open Quant Pad.
 - 9.6.2. Click “Method” button on the Quant Pad button panel.
 - 9.6.2.1. Click the “Select Method” button.
 - 9.6.2.2. Select the file of the quant method you want to use for the gas cylinder concentration check and then choose “OK”. An analysis method can be made following the procedures described in Section 9.11. If the gas concentration being checked is outside the analysis range, dilute the gas (SOP G11).
 - 9.6.3. Click “Collect” button on the Quant Pad button panel.
 - 9.6.3.1. Set the number of scans for both background and sample at 64.
 - 9.6.3.2. Click the “More” button at the bottom of the above dialog box to check the settings of the data collection parameters.
 - 9.6.3.2.1. Make sure that Resolution, Apodization, Zero Filling, Final Format, and File Handling parameters are set properly according to Table 1.
 - 9.6.3.2.2. If a new background is needed before taking the sample spectra (e.g., when the sample is going to be taken more than 4 h after the last background spectrum is taken), make sure the background setting is placed at the third option (“collect background after x minutes”).
 - 9.6.3.2.3. Otherwise, make sure the background setting is placed at the fourth option (“use specified background file”), and that the latest background file is loaded.
 - 9.6.3.2.4. Click “OK” when finished setting the collection parameters.
 - 9.6.4. Align the optical bench by clicking “Align” in the Quant Pad button panel.
- 9.7. Perform FTIR gas absorption cell leak-check. Follow the procedures described in Section 11.8.
- 9.8. Collecting a background spectrum
 - 9.8.1. Check the settings of the data collection parameters by clicking the “Collection” button on the Quant Pad button panel. Make sure the background setting is placed at “collect background after xx minutes”.
 - 9.8.2. Flush the FTIR chamber with pure nitrogen, delivered directly from gas cylinders, for at least 10 min. Control delivery rate at 5-10 L/min. Continue to deliver nitrogen while the background spectrum is being collected.
 - 9.8.3. Click the “Background” button on the Quant Pad button panel.
 - 9.8.4. Click “OK”.
 - 9.8.5. Wait until the specified number of scans has been collected. A typical background spectrum is depicted in Fig. 1.
 - 9.8.6. Click “Yes” to add the background spectrum to the indicated spectral window.
 - 9.8.7. Use the “Save As” command in the OMNIC File menu to save the background spectrum on the hard disk.
 - 9.8.8. A new background should be taken at least once at the beginning of the day’s work, or preferably every 4 h during continuous scanning.

9.9. Collecting and analyzing a sample spectrum

- 9.9.1. Check the settings of the data collection parameters by clicking the “Collection” button on the on the Quant Pad button panel. Change the background setting to “use specified background file” option, and load the file where the most recent background spectrum is saved. When collecting sample spectra, use the same parameter settings that were used to collect the standard spectra (Table 1). Collect the sample and standard spectra at the same pressure and approximately the same temperature.
- 9.9.2. Introduce sample
 - 9.9.2.1. Batch Sampling (offsite cylinder): Load bag sample into FTIR chamber using vacuum/fill step.
 - 9.9.2.1.1. Purge the FTIR gas cell with nitrogen at 5-10 L/min for at least 10 min.
 - 9.9.2.1.2. Close inlet valve on FTIR.
 - 9.9.2.1.3. Attach vacuum pump to the outlet tubing connected to the FTIR.
 - 9.9.2.1.4. Turn on the vacuum pump, open outlet valve on FTIR, and wait until the pressure drops to stable level (usually around 80-200 torr).
 - 9.9.2.1.5. Close the outlet valve. Turn off vacuum pump.
 - 9.9.2.1.6. Attach sample bag to the inlet tubing. Open the bag valve. Minimize the length of inlet tubing to reduce errors in gas concentrations.
 - 9.9.2.1.7. Open the FTIR inlet valve partially and let the bag sample deliver to the FTIR gas cell, close the inlet valve until the pressure reading stabilizes to ambient pressure. Close the bag valve.
 - 9.9.2.1.8. Repeat Steps 9.9.2.1.3 to 9.9.2.1.7 to deliver and purge the sample 3 times. Use the third-time sample as the representative sample delivered from the bag. Wait for at least 2 min for stabilization of gas delivered to the FTIR gas cell before taking the sample spectrum.
 - 9.9.2.1.9. If the gas concentration from the gas cylinder is greater than the maximum analytical range of the current method, then a new method has to be established by incorporating a standard spectrum with a certified gas concentration greater than the gas cylinder concentration.
 - 9.9.2.2. Continuous Sampling (onsite cylinder): Load sample directly from cylinder into FTIR.
 - 9.9.2.2.1. Follow the procedures described in SOP G2 (Standard Operating Procedure for Compressed Gas Cylinders) to deliver gas directly from the gas cylinder.
 - 9.9.2.2.2. If the gas concentration from the gas cylinder is greater than the maximum analytical range of the method, use Environics (See SOP G11) or TEI 146 diluter (See SOP C3) to dilute the gas concentration to below the maximum concentration level that the method can analyze accurately before taking the spectrum.
 - 9.9.2.2.3. Flush the FTIR gas cell with nitrogen beforehand. Vacuum the FTIR gas cell before delivering standard gas to reduce the time before a stable concentration can be reached. Deliver the sample gas at a flow

- rate of 5 L/min for at least 10 min to ensure delivery of a representative sample of the gas to be checked.
- 9.9.3. Click “Sample” button on the Quant Pad button panel. Scanning of the sample will begin immediately. The final spectrum displays when the specified number of scans have been collected.
 - 9.9.4. Click “Save As” in the OMNIC File menu to save the displayed sample spectrum.
 - 9.9.5. Click the “Quantify” button on the Quant Pad button panel. The Quantify window shows the title of the selected quantitative analysis method and the path length, temperature and pressure values it used to calculate the results, followed by the concentration value and confidence range for each component.
 - 9.9.6. To analyze a spectrum already saved on the disk, click the “Open” button on the Quant Pad button panel. Click the “Quantify” button on the Quant Pad button panel to automatically display the gas sample concentration.
 - 9.9.7. To save the concentration data in a file on the disk, click the “Save” button in the “Quantify” window. The concentration data will be saved in a file named with the current base name and the extension “CNC”.
- 9.10. Calculating the gas cylinder concentration analyzed by the FTIR
- 9.10.1. Following procedures described in Section 9.9, collect spectra for samples from at least three sample bags filled with the specified gas to be checked, if the gas cylinder is offsite. If the sample is continuously delivered directly from a gas cylinder, collect at least three spectra, at an interval of every 2-3 min.
 - 9.10.2. Average the concentration data. Apply dilution factor if diluted sample is used. Compare the averaged value with the specified span gas concentration. The accuracy of the gas cylinder is confirmed if the average measured concentration is within 5% of the specified span gas concentration.
- 9.11. To make a Single Gas Method:
- 9.11.1. Collect calibration standard, a known concentration sample spectrum, using Quant Pad (select any method file) following the procedures described in Steps 9.2 to 9.9. Examples of standard spectra for representative compounds are displayed in Fig. 2.
 - 9.11.2. Click the “Save as Standard” button on the Quant Pad button panel. Click “OK” after entering the spectrum information displayed on the window.
 - 9.11.3. Enter a file name for this standard in the “File Name” box and select the drive and directory where the standards should be stored.
 - 9.11.4. Open Quant Setup.
 - 9.11.5. Choose “New Method” from the “File” menu
 - 9.11.5.1. Select your sample phase as gas and concentration units as ppm.
 - 9.11.5.2. Click “OK”.
 - 9.11.6. Choose “Components” from the “Edit” Menu
 - 9.11.6.1. Type in the component name.
 - 9.11.6.2. Type an abbreviated name in the component code box.
 - 9.11.6.3. Click “OK”.

- 9.11.7. Choose “Standards” from the Edit Menu
 - 9.11.7.1. Click “Browse”.
 - 9.11.7.2. Find the file of the standard spectrum and click “OK”.
 - 9.11.7.3. Once the file is specified to be used in a method, it can not be moved out of its specified directory or the method will fail. If you choose to move the file, the method must be modified to match.
 - 9.11.7.4. Check that the concentration, temperature, and pressure are correct for your standard.
 - 9.11.7.5. Click “OK”.
- 9.11.8. Choose “Regions/Windows” from the Edit Menu
 - 9.11.8.1. Carefully study the calibration spectra and choose frequency ranges in which the component peak are clearly identifiable and can be used to quantify the component in a method. Avoid using regions which show high absorption for water vapor or CO₂. Do not choose a frequency range in which the absorbance units are greater than 1 for the component to be quantified. The recommended regions for certain representative compounds are listed in Table 2.
 - 9.11.8.2. Click the “Region Add” button to add regions.
 - 9.11.8.3. If you want Quant Setup to use only certain windows in this analysis region to calculate component concentrations, click in the “Window” box and then click the “Window Add” button to add windows.
 - 9.11.8.4. Repeat the above two steps until all regions and windows have been specified.
- 9.11.9. Choose “Calculations” from the Edit menu
 - 9.11.9.1. Type an “S” for the region of the spectrum that you wish to use to calculate the concentration of this component. For the other regions, type “I”.
 - 9.11.9.2. Click “OK”.
- 9.11.10. Choose “Save Method As” from the File Menu
 - 9.11.10.1. Enter a title for the method.
 - 9.11.10.2. Click “OK”.
- 9.12. Validating the Method:
 - 9.12.1. Once Quant Pad is running, select any method and collect at least one sample spectrum from a sample containing a known concentration of the component, using Quant Pad and the method described above. The concentration should be about one-tenth of the concentration of the corresponding standard used to make the above method.
 - 9.12.2. Save spectrum as a standard with Quant Pad.
 - 9.12.3. Create a validation sample set
 - 9.12.3.1. From Quant Setup, choose “Sample Sets” from the Analyze menu.
 - 9.12.3.2. Click “New”.
 - 9.12.3.3. Click “Add” button.
 - 9.12.3.4. Select file names of spectra you wish to add.
 - 9.12.3.4.1. To change the information associated with the file, click the file name, then click in the appropriate text box and enter the correct value.

- 9.12.3.4.2. To display a spectrum, click “View Spectra” in the Samples dialog box.
- 9.12.3.5. Click “OK”.
- 9.12.3.6. Click “Save Set”.
- 9.12.3.7. Enter a unique title for the sample set.
- 9.12.3.8. Click “New” to add this sample set to the list of sample sets.
- 9.12.3.9. Click “OK” to close the Samples dialog box.
- 9.12.4. Quantify the validation sample set
 - 9.12.4.1. Be sure the correct method is open.
 - 9.12.4.2. Choose “Quantify” from the Analyze menu.
 - 9.12.4.2.1. This will open a dialog box containing information about the sample set based on the method selected.
 - 9.12.4.2.2. The calculated component concentrations will be displayed.
 - 9.12.4.2.3. Click the next button to see results for the next sample in the set.
 - 9.12.4.3. When finished click “OK”.
 - 9.12.4.4. If the calculated component concentrations differ from the actual concentrations by more than an acceptable amount (usually 1 to 5%, depending on the application), and if the errors tend to change regularly as a function of concentration, the absorbance vs. concentration plot for that component may be curved instead of linear. In this case, follow the instructions below to calculate a correction curve for that component.
- 9.12.5. Calculate a correction curve:
 - 9.12.5.1. Use Quant Pad Application to collect spectra from different known concentrations (preferably 4 to 6 different concentration levels) and save as standards using Quant Pad.
 - 9.12.5.2. Open “Quant Setup” and click “Open Method” from the File menu.
 - 9.12.5.3. Select the title of the method to be corrected in the drop down list box and choose “OK”.
 - 9.12.5.4. Create the calibration sample set:
 - 9.12.5.4.1. Choose “Sample Sets” from the analyze menu.
 - 9.12.5.4.2. Click “New”.
 - 9.12.5.4.3. Click the “Add” button.
 - 9.12.5.4.4. Select file names of the spectra you want to add.
 - 9.12.5.4.5. Check to be sure information for each spectrum is correct.
 - 9.12.5.4.6. Click “OK”.
 - 9.12.5.4.7. Click “Save Set”.
 - 9.12.5.4.8. Enter a unique title for your new sample set.
 - 9.12.5.4.9. Click “New” to add this sample set to the list of sample sets.
 - 9.12.5.4.10. Click “OK” to close the Sample dialog box. This set automatically becomes the active sample set.
- 9.12.6. Calculate the correction curve coefficients and save the calibration sample set.
 - 9.12.6.1. Click “Calibration” from analyze menu.
 - 9.12.6.2. Select the component you want to correct by clicking its name in the “Component” list.

- 9.12.6.3. Press the correction button in the calibration dialog box to analyze the calibration sample set using the primary standard for the selected component.
 - 9.12.6.3.1. To force the curve through zero, turn on the “Force Zero Intercept” Option. The accuracy of the method can be improved by forcing the correction curve through zero if the concentration of the analyte in the samples may approach zero. Do not force the curve through zero if the lowest expected concentration in the samples is higher than the lowest standard in the calibration set, especially when all of the spectra in the calibration set are taken at high concentration range.
- 9.12.6.4. Calculate the correction curve coefficient.
 - 9.12.6.4.1. Click on the “Calculate” button in the calibration dialog box to calculate the correction curve coefficients.
 - 9.12.6.4.2. To exclude a selected calibration spectrum from the curve calculation (in case the spectrum is suspected to be an outlier or out of the desired calibration concentration range), select its file name and then turn off the “Include This Sample” option. If necessary, re-run a standard spectrum at such concentration to guarantee the accuracy of the standard spectrum concentration, and reintroduce the new spectrum into the calibration sample set.
 - 9.12.6.4.3. Click “Display” in the Correction Curve Selection dialog to see a graph of the actual vs. measured values.
 - 9.12.6.4.4. Click “Results” in the Correction Curve Selection dialog to see detailed information about the fit of the selected polynomial function to the spectral data.
 - 9.12.6.4.5. Click the up or down arrow button next to the selected order to examine a higher- or lower-order function.
 - 9.12.6.4.6. Select the lowest curve order that fits the actual concentration to the calculated concentrations with the required degree of accuracy specified in the QAPP.
- 9.12.6.5. Transfer curve coefficients to the current method.
 - 9.12.6.5.1. Display the Correction Curve Selection dialog box and make sure the correct polynomial order is selected.
 - 9.12.6.5.2. Click the “Use” button. This transfers the current set of coefficients into the current method for use in correcting the concentrations of the selected component.
- 9.12.6.6. Save the Calibration sample set.
 - 9.12.6.6.1. Press the “Return” button in the Correction Curve Selection dialog box. Calibration Data dialog box appears on screen.
 - 9.12.6.6.2. Press “Save Set”, then either click “Replace” or enter new title and click “New”.
 - 9.12.6.6.3. Press “Return” to get to Quant Setup menu.
- 9.12.7. Save the corrected method by clicking “Save Method” from File menu, or clicking “Save Method As” and enter a title for the method, and then choose “OK”.

- 9.12.8. To run the corrected method, open Quant Pad and select the method. Then either open, quantify, or collect and quantify a spectrum.
- 9.13. Troubleshooting
- 9.13.1. Troubleshooting measures for software, hardware, and application problems are listed in the last three chapters of the ThermoNicolet Nexus 670 User's Guide.
- 9.13.2. If the above action does not solve the problem, call Thermo Nicolet Service office at 1-800-642-6538, 608-276-6100, or email them at techinfo@thermonicolet.com.

10. Data and Records Management

- 10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a record book designated for the method, which should contain copies of the electronic record.
- 10.2. Manage all data according to SOP B5.
- 10.3. Document all data and information (e.g., sample collection method used) on field data sheets, and within site logbooks with permanent ink, or in electronic field notes.
- 10.4. Maintain gas certification records.
- 10.5. Store the quantitative analysis method and spectra (standard, background, and sample spectra) on a computer disk. Store a second backup copy on a separate disk.

11. Quality Control and Quality Assurance

- 11.1. The accuracy of any FTIR quantitative analysis is limited by the accuracy of the concentrations of the gas standards used in establishing the calibration method. Preferably the calibration gas should be certified using EPA protocol (1% certification).
- 11.2. The calibration spectra must be of pure components, or pure components diluted with a solvent or gas that either doesn't absorb infrared energy or absorbs in a different region of the infrared spectrum.
- 11.3. Check the FTIR calibration on a monthly basis by comparing the concentrations of the spectra taken from a reference span gas cylinder with its specified gas cylinder concentration that is guaranteed of accuracy. Recalibrate the instrument if the average measured concentration deviates more than 5% from the specified span gas concentration.
- 11.4. New background spectra should be collected regularly, preferably once every 4 h during the operation of the FTIR.
- 11.5. Align the spectrometer daily for optimum performance.
- 11.6. Keep the room temperature where the FTIR is installed at temperatures between 16°C and 27°C.
- 11.7. Verify that the detector is cooled by liquid nitrogen before taking any spectrum.

- 11.8. Perform FTIR gas absorption cell leak-check before taking sample spectra. Leak check the FTIR cell under both vacuum and pressurized (greater than ambient) conditions.
 - 11.8.1. For the batch sampling technique, close the inlet valve to the FTIR cell, open the outlet valve and connect to a vacuum pump. Evacuate the absorption cell to the minimum absolute pressure P_{\min} . Close the outlet valve, and determine the change in pressure P_v after 2 min.
 - 11.8.2. For both the batch sampling and continuous purging techniques, pressurize the system to about 100 mmHg above atmospheric pressure. Determine the change in pressure P_p after 2 min.
 - 11.8.3. Determine the percent leak volume $\%V_L$, calculated as P_{\max} divided by atmospheric pressure and then multiplied by 100%, where P_{\max} is the larger of P_v or P_p .
 - 11.8.4. Leak volumes in excess of 4% of the FTIR system volume are unacceptable, and the tubing connection should be re-checked.
- 11.9. Prior to use of sample bags for storage of gas samples for subsequent FTIR analysis, perform tests to evaluate sample recoveries and/or contamination from sample bags under controlled conditions. This could be accomplished by comparing FTIR responses to gases in the purge mode and in the batch mode simulating bag materials (walls and fittings), storage times and storage temperatures. Choose the type of sample bag that has the least amount of gas loss and contamination.

12. References

- 12.1. EPA Test Method 320. Measurement of Vapor Phase Organic and Inorganic Emissions by Extractive Fourier Transform Infrared (FTIR) Spectroscopy. www.midac.com/apnotes/Ap-139.PDF
- 12.2. Thermo Electron Corporation. 2001. Thermo Nicolet Nexus 670 User's Guide. Madison, WI.
- 12.3. Thermo Electron Corporation. 2002. Thermo Nicolet Spectrometer Safety Guide. Madison, WI.
- 12.4. Thermo Electron Corporation. 2002. Thermo Nicolet OMNIC User's Guide. Madison, WI.
- 12.5. Nicolet Instrument Corporation. 1995. QuantPad User's Guide. Madison, WI.
- 12.6. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 12.7. SOP G11. 2006. Operation of the Environics® Computerized Gas Dilution System. Standard Operating Procedure G11. Purdue Ag Air Quality Lab.
- 12.8. SOP G2. 2006. Compressed Gas Cylinders. Standard Operating Procedure G2. Purdue Ag Air Quality Lab.

13. Contact Information

13.1. Technical Support: 1-800-Nicolet (1-800-642-6538), 1-608-276-6373.

13.2. PAAQL: odor@purdue.edu, heber@purdue.edu, <http://www.AgAirQuality.com>.

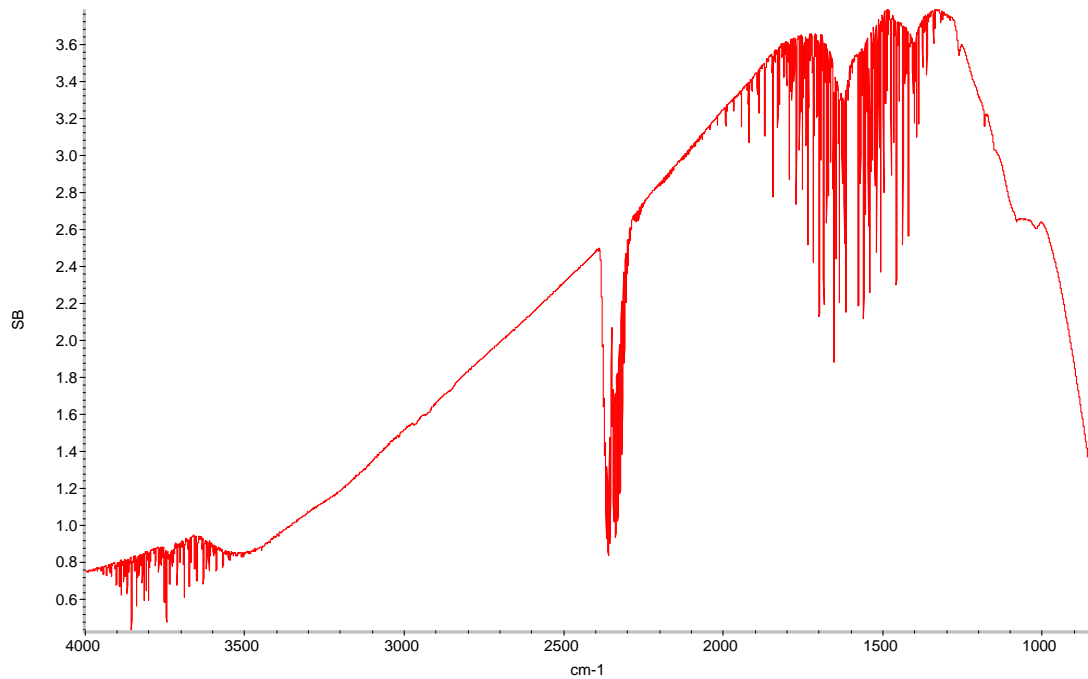


Figure 1. Example of a background spectrum.

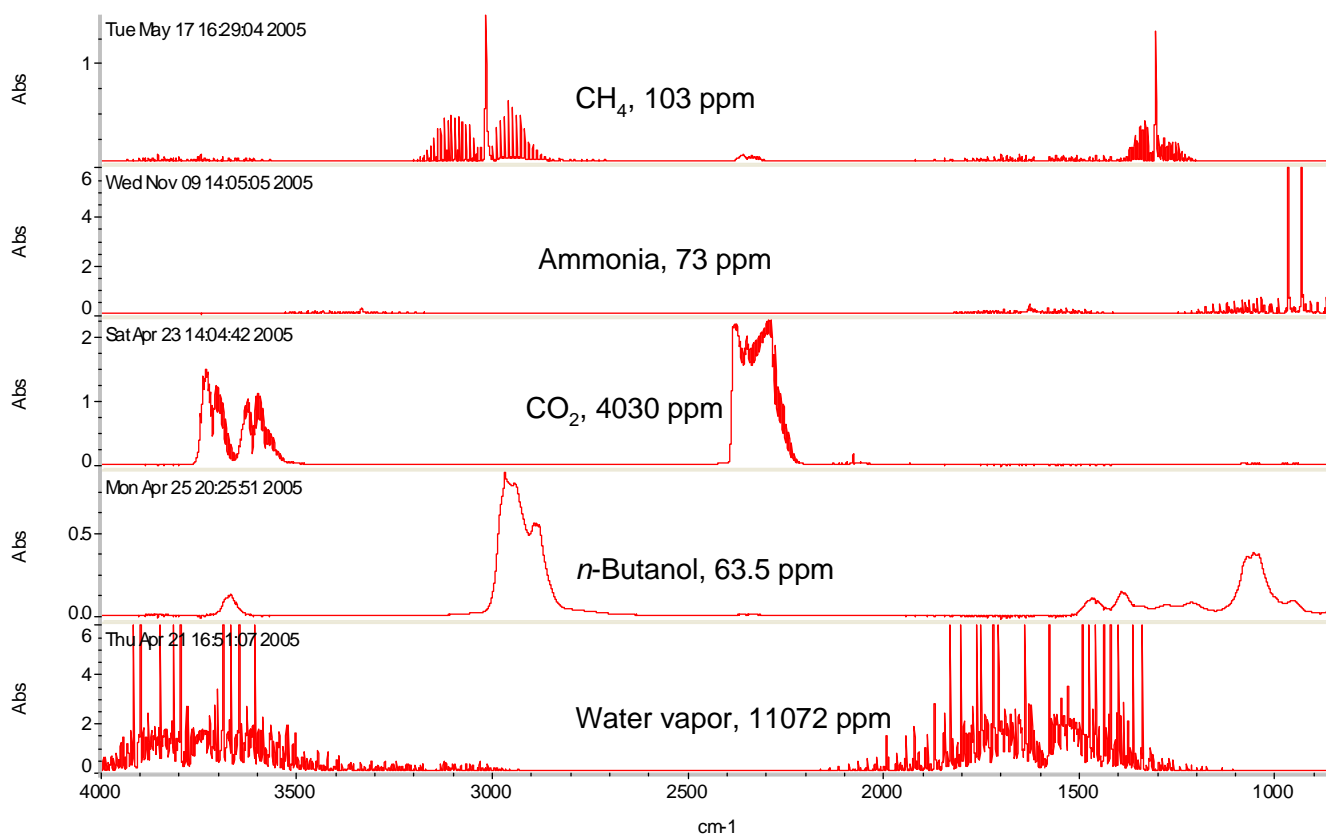


Figure 2. Standard spectra for representative compounds.

Table 1. Data collection and optical bench setup parameter for standard and sample spectra collection.

Parameter	Setting
Number of sample scans	64
Number of background scans	64
Resolution	1
Data spacing	0.482 cm-1
Spectral range	4000-850 cm-1
Mirror velocity	1.8988
Gain	1
Aperture	34
Level of zero filling	0
Apodization	Happ-Genzel
Phase correction	Mertz

Table 2. Preferred frequency ranges to be used as calculation regions for making the analysis methods for each compound.

Compound Name	Frequency Ranges (cm⁻¹)
Carbon dioxide	2075.7-2080
Water vapor	3372.7-3405
Methane	3008.2-3019.6
n-Butanol	2809-3019
Ammonia	961.3-969

**MEASUREMENT OF LAGOON SLUDGE DEPTH
WITH MARKLAND MODEL 10 PORTABLE SLUDGE GUN
Standard Operating Procedure (SOP) L5**

**MEASUREMENT OF LAGOON SLUDGE DEPTH
WITH MARKLAND MODEL 10 PORTABLE SLUDGE GUN
Standard Operating Procedure (SOP) L5**

Prepared by

Chris D. Fullerton and Richard H. Grant

Reviewed by

Bill W. Bogan and Albert J. Heber

Effective Date: November 6, 2006

PURDUE APPLIED METEOROLOGY LABORATORY (PAML)

Agronomy, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability.....	3
2. Summary of Method.....	3
3. Definitions.....	3
4. Health and Safety.....	3
5. Cautions.....	4
6. Interferences.....	4
7. Personnel Qualifications.....	4
8. Equipment and Supplies.....	5
9. Procedures.....	5
10. Data and Records Management.....	7
11. Quality Control and Quality Assurance.....	7
12. References.....	8
13. Contact Information.....	8
Appendix A. Markland Model 10 Sludge Gun Calibration Form.....	9
Appendix B. Sludge Depth Field Data Form.....	10
Appendix C. Markland Sludge Gun Operating Instructions.....	11
Appendix D. Depth Plot Worksheet.....	13

- 4.1.4. Do not attempt to take measurements from the lagoon or board the sampling boat if threatening weather is nearby or imminent.
- 4.1.5. Use safety glasses when near the lagoon water, and take care not to splash lagoon contents on clothes.
- 4.1.6. Use rubber gloves when handling the Markland sludge gun, the boat, or any other gear that may have come in contact with the lagoon contents.
- 4.2. Wash and dry gloves before storing
- 4.3. Wash hands after working in the lagoon or on anything that may come in contact with lagoon contents.
- 4.4. Notify your field partner and apply decontamination and correct field dressing if you accidentally cut yourself or puncture the skin in any manner while handling the sludge gun or any material that may have come into contact with the lagoon contents.
- 4.5. It is recommended to wait until close to the end of the day to take measurements from the lagoon if possible so you can proceed to change clothes and shower as soon as possible afterwards.
 - 4.5.1. Have a spare change of clothes at the site with you at all times.
- 4.6. If you fall into the lagoon or become soaked with fluids from the lagoon:
 - 4.6.1. Immediately notify your partner.
 - 4.6.2. Immediately wash your face and hands.
 - 4.6.3. Locate shower facilities and change clothing if your skin has become soaked.
 - 4.6.3.1. If needed, arrange for transport to your hotel to change clothes and shower, return to work site as soon as possible afterwards.
 - 4.6.3.1.1. Place plastic sheeting on the seats and floor of the work vehicle to avoid contaminating them.

5. Cautions

- 5.1. Twisting the cable will damage it.
- 5.2. If the probe is to be lowered in the vicinity of any moving rakes or scrapers, make sure these are stopped first.
- 5.3. Unwind and rewind cable slowly, to avoid stirring up a large 'cloud' of sludge, which would result in inaccurate readings.
- 5.4. Never drop the probe into any tank with a hard (e.g. concrete) bottom.

6. Interferences

- 6.1. Battery life is shortened in cold weather. For accurate readings, use new batteries during winter operation (i.e. if temperature is near or below freezing).

7. Personnel Qualifications

- 7.1. Each member of the field staff must read and understand this SOP before operating this instrument.

8. Equipment and Supplies

- 8.1. Model 10 Sludge Level Detector (Markland Specialty Engineering, Toronto, Canada)
- 8.2. Cloth and rubbing alcohol
- 8.3. Dish soap
- 8.4. 500-mL beaker to serve as a calibration chamber
- 8.5. Water softener resin
- 8.6. 50-ft tape measure with metric markings
- 8.7. 1-m ruler with float
- 8.8. 10-cm ruler
- 8.9. Heavy duty rubber gloves (2 pair)
- 8.10. (2) Safety glasses
- 8.11. (2) Life vests
- 8.12. 300-ft Life-line rope capable of lifting 300 lbs.
- 8.13. Roll of plastic sheeting (for protecting vehicle from contamination with fecal matter should transport be necessary with contaminated clothing)
- 8.14. AA alkaline batteries (sensor uses 6)
- 8.15. Heavy duty two man inflatable raft with oars
- 8.16. Portable 12-VDC inflation unit.

9. Procedures

- 9.1. Acceptance
 - 9.1.1. Unpack the unit and check for visible damage or missing parts.
 - 9.1.2. Clean the cable using the following method.
 - 9.1.2.1. Unreel all the cable from the probe and lay it out in a straight line on a clean grassy area. Wipe the entire cable with a clean cloth saturated with rubbing alcohol. Allow a few minutes for the cable to dry.
 - 9.1.3. Make sure the cable is clean and dry. Starting at the sensor end and working towards the “gun” body, string a 50-ft. tape measure beside the cable, with the zero end of the tape at the sensing window in the probe gap (see Fig. 1). Peel off the numbers provided with the instrument from the manufacturer and place the “1” at the 1-m mark on the cable, “2” at the 2-m mark, etc.
 - 9.1.4. Calibrate sensor according to Section 9.2
 - 9.1.4.1. If the sensor cannot be calibrated to within the accuracy and precision limits given in Sections 1.3.2 and 1.3.3, return sensor to manufacturer for repair or replacement.
- 9.2. Calibration
 - 9.2.1. Set sensitivity on the Sludge Level Detector in midpoint of range, using the knob on the back of the handle (Fig. 1). Rotate the control knob so that, with the trigger depressed, there is no sound when the probe is in air or in the liquid at the top of the tank, and the high-pitched note sounds when the probe is in the sludge at the lagoon bottom.
 - 9.2.2. Add 200 mL of resin and 200 mL of tap water to the 500-mL beaker.

- 9.2.3. Record water depth (in mm) to top of resin on calibration form (Appendix A).
- 9.2.4. Clamp sensor portion of the instrument to a stand at a height of < 1 m.
- 9.2.5. Place beaker below sensor.
- 9.2.6. Lower probe into water until sensor speaker makes a sound.
- 9.2.7. Holding the cable at the point where it reaches the water line, remove cable and record length of cable from waterline to sensor on calibration form.
- 9.2.8. Repeat Sections 9.2.4 through 9.2.8 eight additional times.
- 9.2.9. Calculate precision and accuracy from the 9 sets of measurements as indicated on the calibration form (Appendix A).
- 9.3. Determining average depth of sludge and water for the lagoon
 - 9.3.1. Refer to Depth Plot Worksheet (Appendix D) for proper sampling locations.
 - 9.3.1.1. Visualize a grid over the lagoon to be measured consisting of intersecting points by making vertical lines from one edge of the lagoon to the other, at approximately the $\frac{1}{4}$ point the $\frac{1}{2}$ way point and the $\frac{3}{4}$ point, and doing the same in the horizontal direction.
 - 9.3.2. Switch gun on. A short 'beep' indicates nominal operation.
 - 9.3.3. Proceed to the first sampling point on the grid.
 - 9.3.4. To unwind or rewind cable, point the gun downward and rotate the whole gun in a circular motion. The cable will come off the front of the spool. Unwind only as much cable as needed, then pass it through the pinch-groove at the front of the gun (Fig. 1). To get the cable back up onto the spool, guide the cable with one hand and rotate the whole gun with the other, so the cable comes back onto the spool.
 - 9.3.5. Slowly lower the probe while squeezing the probe trigger.
 - 9.3.5.1. If the sensitivity control is set correctly, there will be no sound.
 - 9.3.5.2. If sound is heard, adjust sensitivity according to Section 9.2.1.
 - 9.3.5.3. As the probe nears the sludge bed, the sensor emits a low-pitched sound.
 - 9.3.5.4. The volume and pitch will increase as the solids concentration increases.
 - 9.3.5.5. Keep the trigger depressed and slowly lower the probe to the bottom of the lagoon (the cable will go slack when on the bottom).
 - 9.3.5.5.1. Record the first cable marker number above the waterline on the Depth Plot Worksheet as value "A" for the current location shown on the worksheet.
 - 9.3.5.5.2. Using the one-meter ruler, measure the distance from this marker to the waterline, and record as value "B".
 - 9.3.5.6. Slowly raise the sensor probe cable until the sound just stops.
 - 9.3.5.6.1. Record the first cable marker number above the waterline as value "C"
 - 9.3.5.6.2. Using one-meter ruler, measure distance from marker to waterline, and record as value "D".
 - 9.3.6. Repeat Section 9.3.5 for each additional required measurement location, filling in all values on the Depth Plot Worksheet.

- 9.3.7. Determine averages for values A, B, C, and D by adding all the measurements for that value (A, B, C, or D) and dividing by the number of measurement locations (9). Enter the averaged values on the “Sludge Depth Field Data Form” (Appendix B) in the appropriate field. File the completed Depth Plot Worksheet to be scanned and saved according to SOP D1.
- 9.4. Preventive maintenance after each set of measurements at the field site
 - 9.4.1. Clean sensor and sensor cable with water and soap and dry before storage.
 - 9.4.1.1. Clean sludge depth gun by unwinding cable and lay cable out lengthwise in a clean grassy or snowy area and then wipe sensor and cable with cloth saturated with soap and water and rewind cable using a dry clean cloth.
 - 9.4.2. Inspect sensor cable for damage. Repair or replace as necessary to maintain cable integrity and wire continuity.
 - 9.4.3. Store indoors (i.e. in the open-source instrument trailer) when not in use.
- 9.5. Troubleshooting
 - 9.5.1. With the probe in air, if a low-pitched ticking occurs as soon as the trigger is depressed, and does not stop upon turning the sensitivity control, this indicates that either the probe is dirty or the batteries need to be replaced.
 - 9.5.1.1. Clean probe and retest.
 - 9.5.1.2. If ticking persists, replace batteries. Remove two screws and take off rear speaker, pull down on the spring-loaded plunger, and slide out the Battery Pack. Insert new batteries, checking orientation against that shown on the pack.
 - 9.5.2. Repeated constant values indicate sensor failure. Replace sensor.

10. Data and Records Management

- 10.1. Record original calibration and measurement data on appropriate forms (Appendices A and B, respectively), which are kept in a waterproof bound record book designated for lagoon sensors.
- 10.2. Transfer calibration and measurement data to worksheets of the electronic field notes spreadsheet designated for this method.
 - 10.2.1. Reserve one worksheet for calibration data, and one for measurement data.
- 10.3. Document all written data and information in site logbooks with permanent ink.
- 10.4. Overstrike errors in writing with a single line. Initial and date all corrections.
- 10.5. Manage all data is managed according to SOP D1.

11. Quality Control and Quality Assurance

- 11.1. Calibrate (Section 9.2) the sensor quarterly.
- 11.2. Clean sensor and cable (Section 9.4.1) and inspect for damage (Section 9.4.2) after every use.
 - 11.2.1. If damaged, repair wires and encase cable with waterproof heatshrink tubing.

12. References

- 12.1. Sludge Gun Manual. 2006. Markland Model 10 Sludge Gun Operating Instructions. Markland Specialty Engineering, Ltd., Toronto, Canada
- 12.2. SOP D1. 2006. Management of Open-source, Weather, and Lagoon-characterization Data. Standard Operating Procedure D1. Purdue Ag Air Quality Lab/ Purdue Applied Meteorology Lab.
- 12.3. Ullman, J.L. and S. Mukhtar. 2004. Implications on ammonia emissions from dairy facilities using dry-lot versus hybrid (free-stall) housing practices. ASAE Paper #044064. American Society of Agricultural Engineers, St. Joseph, MI.

13. Contact Information

- 13.1. Distributor: Markland Specialty Engineering, Ltd
48 Shaft Road, Toronto, Ontario, Canada M9W 4M2
Tel: 416-244-4980, Fax: 416-244-2287
email: markland@sludgecontrols.com

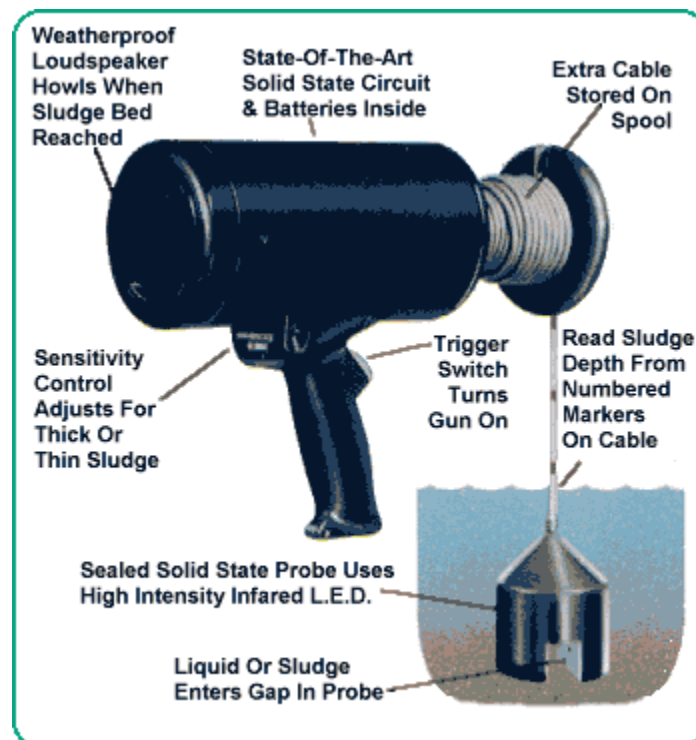


Figure 1. Markland Model 10 Sludge Gun.

Appendix A Markland Model 10 Sludge Gun Calibration Form

Serial Number _____

Date (DD-MM-YYYY)

Person Calibrating _____

Action:

(Accept / Adjust / Replace)

Actual depth of resin layer (mm)	Measured Depth (mm)
	Trial #1
	Trial #2
	Trial #3
	Trial #4
	Trial #5
	Trial #6
	Trial #7
	Trial #8
	Trial #9
Mean	
Standard Deviation (Precision)	
Accuracy (Measured depth – Actual depth)	

Appendix C

MARKLAND SPECIALTY ENGINEERING LTD. **OPERATING INSTRUCTIONS MODEL 10 SLUDGE GUN®**

Cable Markers

Make sure the cable is *clean and dry*. String a 50 ft. tape measure beside the cable, with the zero end of the tape at the Probe. Carefully peel off the numbers and place the “1” at the 1ft. mark, “2” at the 2ft. mark, etc... Metric or any other units can also be used. Placing a strip of black vinyl electrical tape midway between the number markers permits more precision in reading the depth.

Sensitivity Adjustment

This thumb adjustable control is at the back of the handle. Rotate the control knob so that with the trigger depressed, there is no sound with the Probe in air or when it is in the liquid at the top of the tank, and the high pitch note sounds when the probe is down in the sludge. This setting is not critical. The sensitivity control is mainly needed for extremely thick or thin slurries, or for boosting the power as the batteries get weak.

Unwinding/Rewinding Cable

Point the Gun downward and rotate the whole Gun in a circular motion and the cable will come off the front of the spool like a fishing line coming off a reel. Unwind only as much cable as needed, then pass it through the pinch- groove at the front of the Gun. To get the cable back up onto the spool, guide the cable with one hand and again rotate the whole Gun with the other hand so the cable comes back onto the spool. ***Do not twist the cable!***

Finding The Sludge Blanket

Lower the Probe while squeezing the trigger. If the Sensitivity control is set right (see Sensitivity Adjustment) no sound will be heard. As the Probe nears the sludge bed a low pitch sound will come from the speaker. This will increase in volume and rise in pitch as the solids concentration increases. Keep the trigger depressed and lower the Probe to the bottom of the tank (the cable will go slack when on the bottom). Read the cable marker opposite the handrail (for example - 25ft.). The speaker will still be howling with the trigger depressed and the Probe in the sludge. *Slowly* raise the cable until the note starts to become lower pitched, and again read the marker opposite the handrail (for example - 20 ft.). Continue *slowly* raising the Probe until the sound stops and read the cable marker (for example - 18 ft.). In this example, you would have 5ft. of dense sludge, covered by 2 ft. of a partially settled sludge ‘cloud’. Remember to move *slowly*, or you will stir up a large ‘cloud’ and confuse the reading.

Battery Replacement

Batteries will last about one year in normal service. With the Probe in air, if a low pitched ticking is heard as soon as the trigger is depressed, and this cannot be removed by turning the sensitivity control, this indicates that either the Probe is dirty and needs washing, or the batteries are weak and will soon need to be replaced. Ignore the short beep that is heard when the Gun is first switched on. This is normal. Battery life is shorter when the Gun is used at low temperatures. To change batteries, remove two screws and take off rear speaker, pull down on spring loaded plunger and slide out the Battery Pack. Replace with AA size Pencil Alkaline batteries, and be careful to install in the direction shown on the Battery Holder.

Appendix C (continued)**MARKLAND SPECIALTY ENGINEERING LTD.**
OPERATING INSTRUCTIONS MODEL 10 SLUDGE GUN®**Cold Weather Operation**

Battery output is greatly reduced at low temperatures. It is suggested that the Gun be fitted with new batteries if it is going to be used at temperatures near or below freezing. The Gun should be stored indoors when not in use.

Probe

The solid-state Probe is sealed and requires no maintenance other than washing. The cable is sealed at the Probe and can't be disconnected. If defective, the entire Probe/Cable assembly must be replaced. Care should be taken not to tangle the Probe in Rakes or Scrapers, which should be stopped before lowering the Probe into the tank. Do not drop the Probe onto the concrete tank bottom, lower it slowly. Quickly dropping the Probe stirs up the sludge bed making readings difficult, and could cause Probe damage.

Appendix D

Depth Plot Worksheet

Location: _____

Date: _____ Operator: _____

Sensor serial number _____

☆ Denotes approximate measurement locations

A _____ B _____ C _____ D _____	A _____ B _____ C _____ D _____	A _____ B _____ C _____ D _____
☆	☆	☆
A _____ B _____	A _____ B _____	A _____ B _____
☆	☆	☆
C _____ D _____	C _____ D _____	C _____ D _____
☆	☆	☆
A _____ B _____ C _____ D _____	A _____ B _____ C _____ D _____	A _____ B _____ C _____ D _____

MANURE SAMPLING
Standard Operating Procedure (SOP) M1

MANURE SAMPLING
Standard Operating Procedure (SOP) M1

Prepared by
Sam M. Hanni and Bill W. Bogan

Reviewed by
Albert J. Heber and Teng Teeh Lim

Effective Date: November 6, 2006
Rev. 1.0 Effective Date: March 17, 2008

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)
Agricultural and Biological Engineering, Purdue University
West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions	3
4. Health and Safety	4
5. Interferences	4
6. Personnel Qualifications	4
7. Equipment and Supplies	5
8. Procedures	6
9. Data and Records Management	8
10. Quality Control and Quality Assurance	9
11. References	9

1. Scope and Applicability

- 1.1. The release of ammonia from manure in livestock barns and storage areas is affected by manure pH, moisture content, and nitrogen and ammonia contents. Manure must be collected to determine these parameters.
- 1.2. Data on nitrogen content of manure at the end of its storage period is also needed to develop a N balance for the facility (SOP S5).
- 1.3. This SOP describes the procedures for collecting manure samples from broiler, layer, dairy, and swine barns, and from manure storage areas, during tests of air emissions.
 - 1.3.1. Manure samples may need to be collected from various types of storage or treatment facilities, including deep pits under buildings, collection channels, drainage pipes, outdoor storage basins, lagoons, and storage piles.
- 1.4. This SOP provides information on selecting sampling locations and methods to capture representative samples.
- 1.5. The sampling methods described in this SOP are adapted from References 11.1 – 11.4.

2. Summary of Method

Different sampling equipment and techniques should be selected based on manure characteristics (liquid or dry samples) and the type of facility that is being sampled. Storage basins and lagoons can be sampled with either a conduit-mounted container sampler, a sampling tube with a stopper, or a telescoping ladle. The ladle is also well-suited to sampling collection channels or drainage pipes. Deep pits under slatted floors can be sampled with a sampling tube with a stopper, or with a manure/sludge sampling pump. Manure storage piles that do not contain significant litter, feathers, or other material can be sampled using a tube corer. If, however, litter and feathers are plentiful, sampling can be conducted using a shovel or spade. Several sub-samples can be taken to produce a composite sample. It is important to take a sufficient number of samples to be representative of the manure storage or treatment unit. The times at which manure is sampled will depend on the data required. For example, if the sample is intended to provide data on initial manure parameters (pH, moisture, N and/or NH₃ content) that may affect emissions, it may be desirable to collect fresh manure. If, on the other hand, the final N content of aged manure leaving a storage facility is needed for an N balance, sampling should coincide with the emptying of the storage unit. Exact sampling times for each site will be specified in the Site Monitoring Plans in the QAPP for the specific project.

3. Definitions

- 3.1. COC Chain of custody
- 3.2. PAAQL Purdue Agricultural Air Quality Laboratory
- 3.3. QAPP Quality Assurance Project Plan

4. Health and Safety

- 4.1. When sampling manure, wear Tyvec suits, shoe covers, and gloves. Wear a respirator to prevent exposure to manure gases.
- 4.2. Sample, mix, and package all samples in a well-ventilated area.
- 4.3. Agitation of manure in storage facilities (e.g. deep-pit systems) may release gases that cause very strong odors, and high concentrations that are a severe (i.e. lethal) health hazard (Lorimor et al. 2004).
 - 4.3.1. If agitation is deemed necessary for accurate sampling, either remove all animals and persons from the facility before beginning agitation, or open or turn on all available ventilation sources (doors, windows, exhaust fans) throughout the agitation and sampling process.
- 4.4. Be aware of slippery surfaces.
- 4.5. When collecting samples from confined storage spaces, make sure there is someone watching from a safe area.
- 4.6. Do not attempt to take samples from manure piles that are too moist and soft to walk on. If the manure becomes too soft, wait to take samples until it is dry enough to walk on, or until it is removed from the building.
- 4.7. Keep manure sampling equipment and samples away from your mouth and face.
- 4.8. Be aware of aggressive livestock that may interfere with the sampling process.
- 4.9. If animals are interfering with the sampling procedure at a particular location, contact the farm manager so that the animals can be temporarily removed from the location.

5. Interferences

- 5.1. Clean all equipment thoroughly between samplings and sampling locations, using a scrub brush and a bucket of hot, soapy (antibacterial dish soap) water. Check all equipment visually and make sure there is no remaining manure attached.
- 5.2. Nutrient stratification can occur in non-agitated basins, pits, and lagoons. In these cases, it can be particularly important to collect subsamples from as wide a range of depths as possible, so they can be composited to obtain samples that are representative of the entire vertical profile.

6. Personnel Qualifications

- 6.1. Personnel should read and understand this SOP and be trained in the use of sampling equipment.
- 6.2. Personnel should be trained in proper handling and labeling of samples, and in the storage and shipping process.
- 6.3. The individual taking samples should be able to locate the place from which samples are to be taken.

7. Equipment and Supplies

- 7.1. Conduit-mounted container sampler (Fig. 1) for sampling basins and lagoons
 - 7.1.1. The container sampler consists of a long conduit, with a handle at one end and a container with a spring-loaded lid on the other. A cable is attached to the lid of the container, and runs up the conduit and through the handle. When pulled, the cable opens the lid of the container; when released, the spring closes the lid. This type of sampler is particularly useful for collecting samples below the surface of a storage basin or lagoon, if concurrent collection of the surface layer is not required.
- 7.2. Sampling probe or tube (Fig. 2) for sampling lagoons or deep pits
 - 7.2.1. Make the sampling tube from a PVC tube.
 - 7.2.2. For sampling a deep pit, choose a sampler that has an outside diameter smaller than the slot opening of the slats.
 - 7.2.3. For sampling open pit or lagoon, use a tube with a larger outside diameter (up to 6") to reduce the sampling time.
 - 7.2.4. The sampling tube is equipped with a string running the length of the tube. One end of the string is attached to a rubber stopper or ball with a diameter larger than that of the tube; on the opposite end, the string is attached to the sampling tube.
- 7.3. Telescoping sampler for sampling collection gutters or channels
 - 7.3.1. CONBAR telescoping dipper, Enviro-Tech Services, Martinez, CA (or equivalent)
 - 7.3.2. Sampler consists of three nested 4.5' metal conduit sections, which telescope out to a total length of 12'. The end of the sampler has a screw-tight mounting for a 600-mL polypropylene beaker.
- 7.4. Manure/sludge sampling pump and collection system for sampling deep pits under slatted floors
 - 7.4.1. Pump: ¼-horsepower, Gast Model 0523-V191Q-G588DX, or equivalent
 - 7.4.2. Erlenmeyer filtering flasks (2) 2-L or larger. Polypropylene is preferred over glass for safety reasons.
 - 7.4.3. Stoppers and tubing to connect the two flasks in series
 - 7.4.4. Sampling tubing – wall of tubing must be thick enough to withstand collapse under the vacuum from the pump.
 - 7.4.5. PVC pipe or similar rigid support to guide tubing into the pit and allow sampling at depth
 - 7.4.6. Cable ties
- 7.5. Metal tube corer (Fig. 3) for sampling manure piles
 - 7.5.1. The corer consists of a thin-walled metal tube with a solid metal handle.
- 7.6. Sledgehammer
- 7.7. Sharp spade for sampling of feather-containing manure piles
- 7.8. Five-gallon buckets, hand plungers, and mixing rods for sample homogenization
- 7.9. Tyvec suits, shoe covers and gloves
- 7.10. Bucket, scrub brush and antibacterial dish soap
- 7.11. Freezer for storage of manure samples prior to shipping
- 7.12. Shipping materials
 - 7.12.1. Insulated cooler
 - 7.12.2. One-quart plastic bottles or equivalent

- 7.12.3. 8" x 10" and 9" x 12" zip-lock plastic bags
- 7.12.4. Moisture-proof labels for sampling containers
- 7.12.5. Shipping peanuts and/or expandable shipping foam
- 7.12.6. "Blue ice" or other cooling agent
- 7.12.7. Duct tape or packing tape
- 7.12.8. Shipping and chain-of-custody (COC) forms

8. Procedures

- 8.1. Sampling basins or lagoons with container sampler
 - 8.1.1. Immerse container portion of sampler below the surface of the basin.
 - 8.1.2. Pull cable to open container lid and allow sample to fill the open container, then release to close. Avoid collecting floating debris, as these may bias the analysis.
 - 8.1.3. Transfer collected sample into a five-gallon bucket.
 - 8.1.4. Take multiple samples in order to make a composite sample.
 - 8.1.4.1. If the composite sample is intended to represent the entire basin or lagoon, take each individual sample at a different location.
 - 8.1.4.2. If the sample is intended to represent fresh manure, sample from the inlet as manure is entering the basin or lagoon.
 - 8.1.4.3. The total number of samples needed from each basin or lagoon, and the number of different locations needed, will be specified in the QAPP for a specific project.
 - 8.1.5. Mix the composite sample with a hand-held plunger (using an up-and-down motion) until it is well-mixed.
 - 8.1.6. While mixing is maintained, withdraw sample using a cup or similar instrument.
 - 8.1.7. Fill a one-quart plastic bottle not more than three-quarters full and cap tightly.
 - 8.1.8. Log the sample (Section 10.1) and follow the shipping procedures in Section 8.7.
- 8.2. Sampling deep pits or lagoons with sampling tube
 - 8.2.1. Insert sampling tube into the lagoon or deep pit until touching the bottom, with the stopper open.
 - 8.2.2. Pull string attached to stopper to seal the end of the sampling tube. Retract the tube, being careful to avoid tipping the tube and spilling the sample.
 - 8.2.3. Release the sample into a five-gallon bucket.
 - 8.2.4. Take multiple samples to make a representative composite sample.
 - 8.2.4.1. If the composite sample is intended to represent the entire deep pit or lagoon, take each individual sample at a different location.
 - 8.2.4.2. If the sample is intended to represent fresh manure, sample from the inlet as manure is entering the lagoon, or from the top layer of the deep pit.
 - 8.2.4.3. The total number of samples needed from each pit or lagoon, and the number of different locations needed, will be specified in the QAPP for the specific project.
 - 8.2.5. Use a mixing rod to stir the contents of the bucket; this will resuspend settled solids.
 - 8.2.6. While agitating the liquid, use a ladle to transfer the sample into a plastic bottle until the bottle is three-quarters full.
 - 8.2.7. Securely fasten the lid onto the container.
 - 8.2.8. Log the sample (Section 10.1) and follow the shipping procedures in Section 8.7.

- 8.3. Sampling lagoons or collection gutters with telescoping sampler
 - 8.3.1. If not already attached, attach the polypropylene beaker to the end of the sampler. Extend the sampler to the desired length, and immerse container portion of sampler below the surface of the manure.
 - 8.3.2. Transfer the collected sample into a five-gallon bucket. Follow Steps 8.2.4-8.2.8 above to collect and mix a composite sample and prepare it for shipping.
- 8.4. Sampling deep pits under slatted floors with a pump
 - 8.4.1. Connect two filtering flasks in series with the sampling pump, as shown in Fig. 4.
 - 8.4.2. Attach the sampling tubing to the PVC pipe or other support using cable ties. Slide the tubing/support into the manure in the pit to the desired depth. Unless it is specifically intended to sample the sludge layer that is most likely present at the bottom of the pit, it may be desirable to avoid lowering the sampling tube all the way to the bottom, as the sludge may block the tubing.
 - 8.4.3. Turn on the sampling pump. Collect approximately 1 L of manure in the first flask (half full). The second flask should serve as a trap to keep manure from entering the pump.
 - 8.4.4. Turn off the pump and disconnect the sample flask. Mix the contents, either with a stir bar or by swirling, and transfer (pour) the sample into a plastic bottle until the bottle is three-quarters full. Alternatively, if the sample is to be composited with others before taking the final sample, follow Sections 8.1.3-8.1.7.
 - 8.4.5. Securely fasten the lid onto the container.
 - 8.4.6. Log the sample (Section 10.1) and follow the shipping procedures in Section 8.7.
 - 8.4.7. Discard the remainder of the manure from the sample flask, and rinse the flask before re-connecting to collect the next sample.
- 8.5. Sampling storage piles with a spade and/or scoop
 - 8.5.1. With a scoop and a bucket, collect samples throughout the manure storage piles. Take multiple samples in order to make a representative composite sample.
 - 8.5.1.1. If the composite sample is intended to represent the entire storage pile, take each individual sample at a different location.
 - 8.5.1.2. If the sample is intended to represent fresh manure, sample from newest portion of the pile (if necessary, consult with farm personnel to identify this portion).
 - 8.5.1.3. The total number of samples needed from each storage pile, and the number of different locations needed, will be specified in the QAPP for the specific project.
 - 8.5.2. Once all of the samples have been collected, lay a piece of clean plastic on the ground and place the samples onto the plastic.
 - 8.5.3. Using a shovel, continuously scoop the outside of the pile to the center of the pile, and mix the center, until the composite pile is thoroughly mixed.
 - 8.5.4. Use a scoop to collect a composite sample from the mixed pile into a one-quart sealable bag. Fill the bag approximately halfway.
 - 8.5.5. Squeeze excess air out of the bag, seal the bag, and place the bag into a second bag, which should be larger than one quart (e.g. 9" x 12") for ease of handling.
 - 8.5.6. Log the sample (Section 10.1) and follow the shipping procedures in Section 8.7.
- 8.6. Use of core sampler for sampling storage pile
 - 8.6.1. Insert the corer into the manure storage pile. If needed, use a sledghammer to pound the tube portion of the corer into the manure, until it touches the bottom of the pile.

- 8.6.2. Remove the core sampler from the manure pile.
- 8.6.3. Using a long rod or broom handle, push sample from corer into a clean bucket.
- 8.6.4. Take multiple samples to make a composite sample. The total number of samples needed from each pile will be specified in the QAPP for the particular project.
 - 8.6.4.1. If the composite sample is intended to represent the entire storage pile, take each individual sample at a different location.
 - 8.6.4.2. If the sample is intended to represent fresh manure, sample from newest portion of the pile (if necessary, consult with farm personnel to identify this portion).
 - 8.6.4.3. The total number of samples needed from each storage pile, and the number of different locations needed, will be specified in the QAPP for the specific project.
- 8.6.5. Once all of the samples have been collected, lay a piece of clean plastic on the ground and place the samples onto the plastic.
- 8.6.6. Using a shovel, break up any chunks that may be present. Continuously scoop the outside of the pile to the center of the pile, and mix the center, until the composite pile is thoroughly mixed.
- 8.6.7. Use a scoop to collect a composite sample from the mixed pile into a one-quart sealable bag. Fill the bag approximately halfway.
- 8.6.8. Squeeze excess air out of the bag, seal the bag, and place the bag into a second bag, which should be larger than one quart (e.g. 9" x 12") for ease of handling.
- 8.6.9. Log the sample (Section 10.1) and follow the shipping procedures in Section 8.7.
- 8.7. Handling and shipping samples
 - 8.7.1. Label all samples with sample location, date and the name of the personnel who collected the samples. Include a completed COC form with each shipment. Enclose COC form in a zip-lock back to keep it dry and clean.
 - 8.7.2. Load all samples into the cooler they will be shipped in. Add the desired amount of blue ice, ice-gel packs, or equivalent. If necessary, add sufficient packing material (such as packing peanuts or expandable packing foam) to keep the samples from shifting. Place the packed cooler in the freezer overnight (at minimum).
 - 8.7.2.1. Make sure that the cooler is not contaminated with chemicals or manure, and that it can be properly sealed. Use duct tape or packing tape to keep the cooler closed during shipping.
 - 8.7.3. Use overnight shipping. Arrange shipping and receiving to ensure that samples are not held over a weekend or holiday, and that they do not arrive at the lab on a weekend or holiday.

9. Data and Records Management

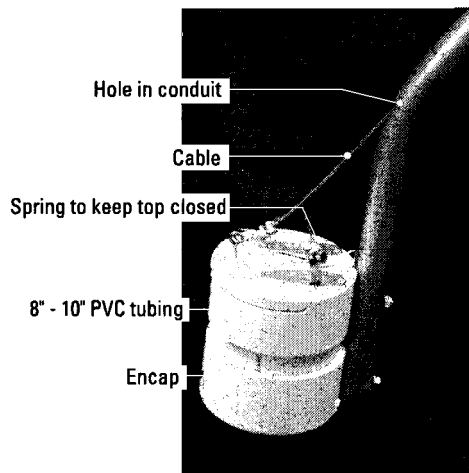
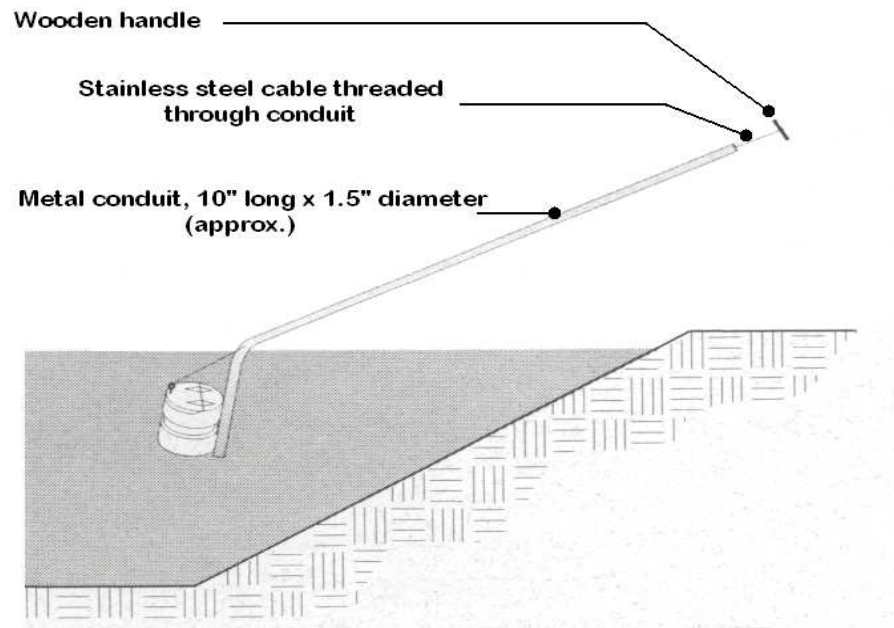
- 9.1. Maintain all records in a worksheet of the electronic field notes spreadsheet which is designated for this method. Supplement this electronic record with a bound record book designated for the method, which should contain copies of the electronic record.
- 9.2. Manage data according to SOP B5.
- 9.3. Document all data and information on field data sheets, and within site logbooks, with permanent ink, or in electronic field notes.

10. Quality Control and Quality Assurance

- 10.1. Log all samples. Identify all collected samples, both on their container and in the site logbook, with the sample name and/or number, location, collection method, and date.
- 10.2. Check all sampling equipment before each sampling trip to make sure they are functioning properly.

11. References

- 11.1. Lorimor, J., W. Powers, and A.L. Sutton. 2004. Manure Characteristics. Manure Management Systems Series #MWPS-18, Section 1. Second Edition. MidWest Plan Service, Ames, IA.
- 11.2. MidWest Plan Service. 1998. Livestock Waste Facilities Handbook. Third Edition. MidWest Plan Service, Ames, IA.
- 11.3. Peters, J., S. Combs, B. Hoskins, J. Jarman, J.L. Kovar, M. Watson, A. Wolf, N. Wolf. 2003. Recommended Methods Of Manure Analysis. American Society for Agronomy-Crop Science Society of America-Soil Science Society of America Annual Meeting Abstracts. University of Wisconsin Extension Service, Madison, WI.
- 11.4. Sonon, L., P. Vendrell, and D. Kissel. Manure and Soil Sampling Analysis. Online at <http://www.engr.uga.edu/service/extension/agp2/resources/presentation/Manure%20and%20Soil%20Sampli179.ppt>. Accessed February 1, 2006.
- 11.5. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.



Pull cable to open top. Release cable to allow spring to close the top before removing container from the liquid.

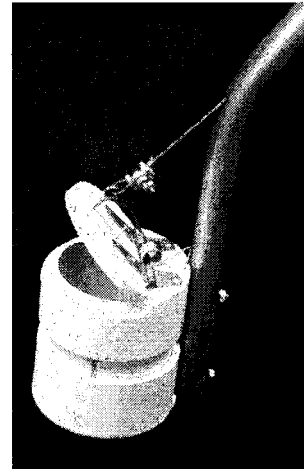


Figure 1. Conduit-mounted container sampler, with close-up showing the operation of the container lid (Lorimor et al, 2004).

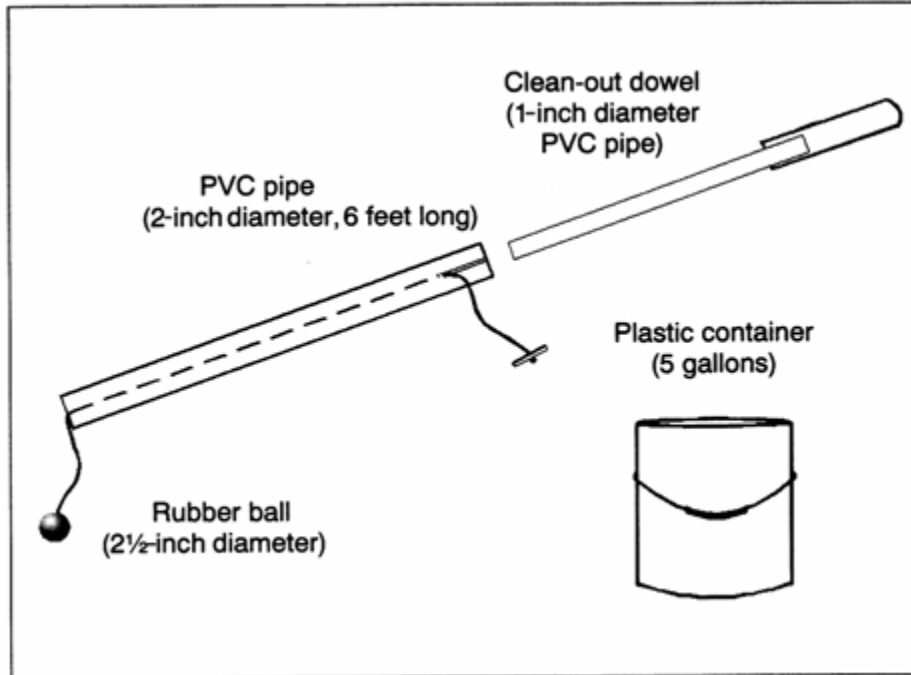


Figure 2. Liquid Slurry Sampling tube (Sonon et al., 2005). Sampler size shown is appropriate for sampling deep pits beneath slatted floors; larger diameters may be used for open lagoons.

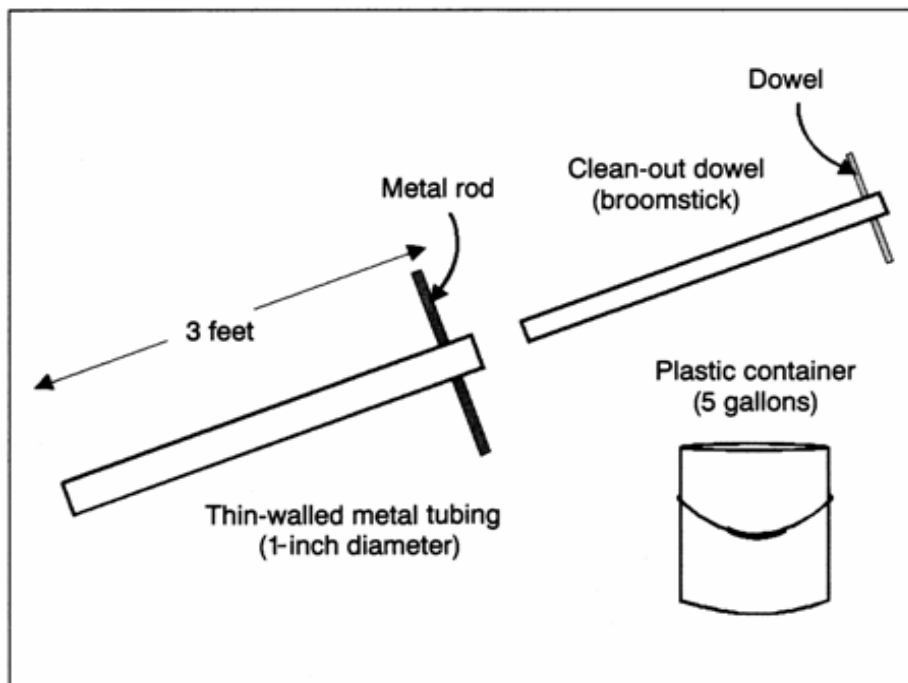


Figure 3. Core Sampler (Sonon et al., 2005).

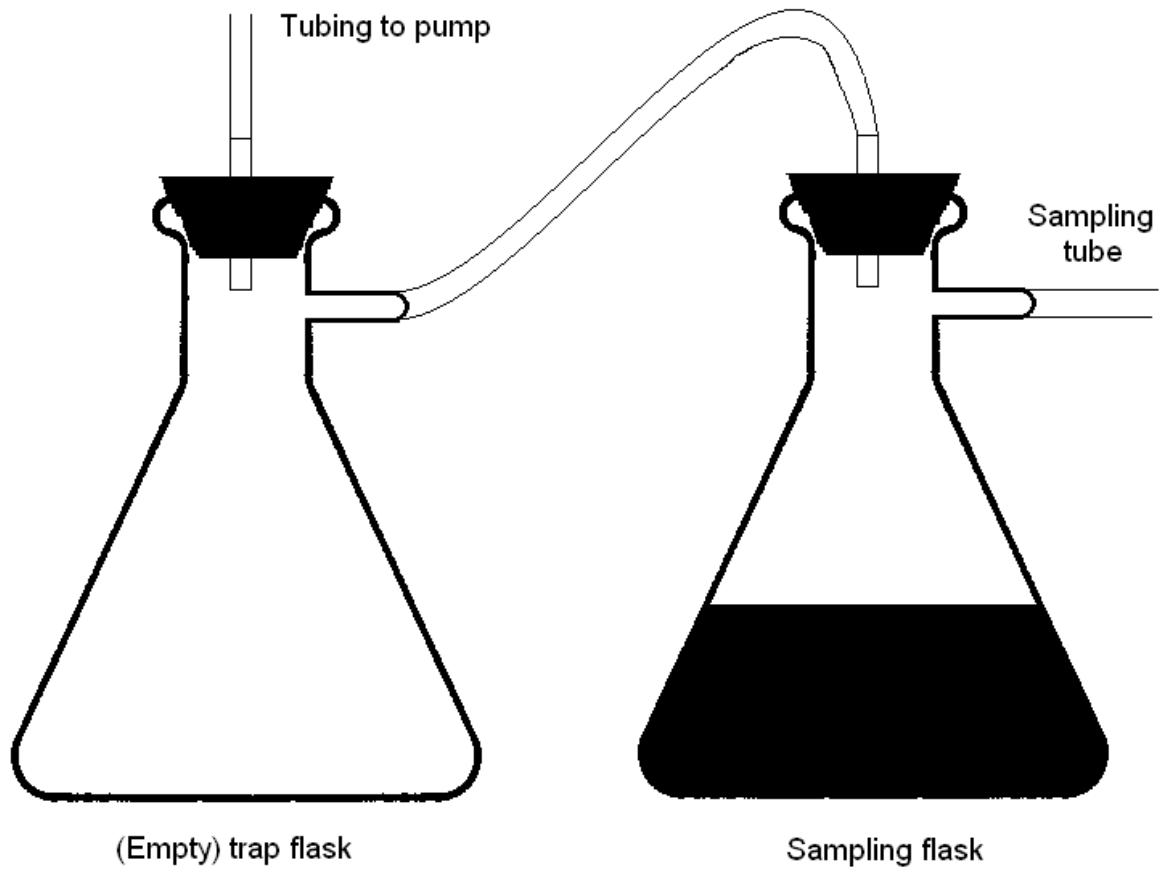


Figure 4. Sampling system for collecting manure from pits under slatted floors (e.g. from deep-pit swine barns).

CONDUCTING pH MEASUREMENTS ON MANURE SAMPLES
Standard Operating Procedure (SOP) M2

CONDUCTING pH MEASUREMENTS ON MANURE SAMPLES

Standard Operating Procedure (SOP) M2

Prepared by

Alan L. Sutton, Brian Richert, Scott Radcliffe, Dan Kelly and Bill W. Bogan

Reviewed by

Albert J. Heber, Harold M. Keener (Ohio State Univ.), and Jerome J. King (Midwest Laboratories, Inc)

Effective Date: November 6, 2006

Rev. 1.0 Effective Date: January 11, 2008

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability.....	3
2. Summary of Method.....	3
3. Health and Safety.....	3
4. Cautions.....	3
5. Interferences.....	4
6. Personnel Qualifications	4
7. Equipment and Supplies	4
8. Procedures	5
9. Data and Records Management	6
10. Quality Control and Quality Assurance	6
11. References.....	6

1. Scope and Applicability

- 1.1. A range of pH-dependent chemical and biological interactions and processes can affect the odor and overall composition of the manure.
 - 1.1.1. Emissions of ammonia (NH_3) increase as pH increases.
 - 1.1.2. Emissions of hydrogen sulfide (H_2S) decrease as pH increases.
- 1.2. The method detailed here is adapted from USEPA Method 9040C, "pH Electrometric Measurement", and is therefore suitable for use on aqueous wastes and those multiphase wastes (including manures) where the aqueous phase constitutes at least 20% of the total volume of the waste. Wastes which originally contain less than 20% water by volume are mixed with water, and allowed to equilibrate, prior to taking the pH measurement.
- 1.3. This procedure measures the pH of a manure sample using the Orion Model 310 pH meter, equipped with a KCl electrode.
 - 1.3.1. This instrument covers the pH range from 0.00 to 14.00.
 - 1.3.2. The instrument has a resolution of 0.01 pH units, and a stated accuracy of ± 0.02 pH units.
 - 1.3.3. The Orion Model 310 can be used within the temperature range of 0°C to 100°C , and includes temperature correction over this range. Its temperature resolution is 0.1°C , with a stated relative accuracy of $\pm 0.5^\circ\text{C}$.
 - 1.3.4. This instrument can be used in laboratory or on-site settings.
 - 1.3.5. This method allows for high-throughput analysis of samples, as analysis of one sample generally requires no more than 5 min.

2. Summary of Method

The pH of the sample is determined electrometrically using either a glass electrode in combination with a reference potential, or a combination electrode. The measuring device (pH meter) is calibrated using a series of standard solutions of known pH, which are chosen such that the calibration range brackets the samples whose pH is to be determined. Liquid samples are measured as collected, while solid samples are equilibrated with deionized water in order to obtain a reading. Because measurement of pH is sensitive to temperature, the Orion Model 310 pH meter and 82-02 electrode are equipped with a temperature-correction capability.

3. Health and Safety

- 3.1. Wear lab coats, gloves and face shields when handling manure samples.
- 3.2. Buffers may cause eye and skin irritations and may be harmful if inhaled or swallowed.

4. Cautions

- 4.1. Avoid rubbing or wiping electrode bulb, as this may introduce error due to polarization.
- 4.2. Do not let stirring bar hit the sensing bulb, as it may crack or break the electrode tip.

5. Interferences

- 5.1. Glass KCl electrodes, in general, are not subject to solution interferences from any of the following factors:
 - 5.1.1. Color
 - 5.1.2. Turbidity
 - 5.1.3. Colloidal matter
 - 5.1.4. Oxidizing or reducing agents
 - 5.1.5. Moderate (<0.1 M) salinity
- 5.2. Coatings of oily material or particulate matter on the electrode surface can impair its response, and should be removed as they arise. These coatings can usually be removed by gentle wiping with detergent, followed by rinsing with distilled water. An additional treatment with hydrochloric acid (1:10) may be necessary to remove any remaining film.
- 5.3. Temperature effects on pH measurement using this method arise from two sources:
 - 5.3.1. Change in electrode output at various temperatures. This interference is controlled by using a pH meter and electrode equipped with temperature compensation.
 - 5.3.2. Change of pH due to temperature-dependent changes in the sample itself. This error is sample-dependent and cannot be controlled. It should, therefore, be noted by reporting both the pH and temperature at the time of analysis.
- 5.4. Manures have considerable bacterial populations, and metabolic acids produced by these bacteria can cause rapid fluctuations (decreases) in pH. Keep manure samples cool during shipping (SOP M1), and freeze upon arrival in the lab to minimize bacterial growth and activity. Analyze samples as soon after thawing as possible. Observe the general holding times described in Section 10.1.3.

6. Personnel Qualifications

- 6.1. Personnel should be trained in the use of the analytical instruments, and in laboratory safety, before initiating the procedure.
- 6.2. Each analyst must read and understand the entire manuals for the pH meter and electrode, and this SOP, before operating the instruments.

7. Equipment and Supplies

- 7.1. Orion Model 310 pH meter (Thermo Electron Corp., Beverly, MA), or equivalent
- 7.2. Orion 82-02 PerpHecT ROSS Combination pH Electrode with automatic temperature compensation (Thermo Electron Corp., Beverly, MA)
- 7.3. Calibration buffers (VWR International, Chester, PA) - These commercially available solutions have been validated by comparison with NIST standards and are recommended by USEPA for routine use.
 - 7.3.1. pH 4 Buffer, Cat. No. 34170-127
 - 7.3.2. pH 7 Buffer, Cat. No. 34170-130
 - 7.3.3. pH 10 Buffer, Cat. No. 34170-133
- 7.4. Magnetic stirrer and Teflon-coated stirring bars

8. Procedures

- 8.1. Select the KCl electrode.
- 8.2. Lower the plastic band covering the hole on the electrode.
- 8.3. Set pH meter to “standby”.
- 8.4. Lift electrode from storage solution.
- 8.5. Using a wash bottle, rinse electrode with redistilled water into a waste beaker.
- 8.6. Instrument calibration
 - 8.6.1. Set meter to “pH” mode.
 - 8.6.2. Press “CAL” key to initiate calibration sequence. “CAL” is displayed for 2 s.
 - 8.6.3. Press the “YES” key to accept the last calibration range (7-4 or 7-10) or select one of these calibration ranges using the SCROLL keys. Press “YES” to accept.
 - 8.6.3.1. When measuring samples with acidic pH, use pH 4 buffer for the second calibration step (Choose 7-4 calibration).
 - 8.6.3.2. When measuring samples with alkaline pH, use pH 10 buffer for the second calibration step (Choose 7-10 calibration).
 - 8.6.4. “pH 7” will be displayed for 2 s. Place electrode in pH 7 buffer, using a sufficient volume of buffer in a 50-mL beaker to cover the sensing elements of the electrodes, and to give adequate clearance for the magnetic stirring bar. Reading will be displayed and updated as calibration continues. When the “READY” light comes on indicating electrode stability, press the “YES” key to accept.
 - 8.6.5. “pH 4” (or “pH 10”) will then be displayed for 2 s. Remove electrode from pH 7 buffer. Rinse with deionized water and place electrode in either pH 4 or pH 10 buffer (depending on calibration range that was selected). When the “READY” light comes on, press the “YES” key to accept buffer value. “SLP” (slope) will be displayed for 2 s, after which the calculated slope will be displayed.
 - 8.6.6. Meter will automatically go into the “MEASURE” mode, at which point the “READY” light will come on to indicate electrode stability.
- 8.7. Using a wash bottle, rinse electrode with redistilled water into a waste beaker.
- 8.8. If sample is a liquid, or a slurry with >20% water by volume, place approximately 30-40 mL of sample to be tested into a beaker and gently stir at a constant rate to provide homogeneity and suspension of solids.
 - 8.8.1. If sample is a solid, or a slurry with <20% water by volume, mix approximately 5 g of sample with water in a 1:1 ratio in a beaker. Gently stir at a constant rate to provide homogeneity and suspension of solids, and allow sample to equilibrate for 30 min before taking the measurement.
- 8.9. Submerge electrode; when readout is stable, note and record sample pH and temperature.
 - 8.9.1. If operating in the LogR mode or using an ATC (Automatic Temperature Compensation) probe, the temperature-corrected pH is displayed.
- 8.10. Rinse electrode between samples with distilled water into the waste beaker.
- 8.11. When finished, submerge electrode in storage solution and return pH meter to “standby”.

9. Data and Records Management

- 9.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
- 9.2. Manage all data according to SOP B5.
- 9.3. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes. Overstrike all errors in writing with a single line. Initial and date all corrections.

10. Quality Control and Quality Assurance

- 10.1. Data integrity
 - 10.1.1. A second person will verify that the analyses were conducted properly and provide a signature at the end of the data set run and/or at the end of the working day.
 - 10.1.2. Record the temperature with each set of pH measurements, in accordance with Section 5.3.2.
 - 10.1.3. In accordance with USEPA Method 9040C, analyze all samples as soon after collection as is feasible.
 - 10.1.3.1. Hold refrigerated samples for no more than 7 days prior to conducting this analysis. Archived (frozen) samples can be maintained indefinitely.
- 10.2. Equipment handling and maintenance
 - 10.2.1. Submerge electrode in storage buffer at all times when not in use.
 - 10.2.2. Maintain the level of filling solution above the reference junction and at least one inch above the sample level when immersed. Add saturated KCl solution to the electrode if it falls below this level.
 - 10.2.3. Calibrate the instrument (See Section 8.6) immediately before each batch of samples, and after each batch as a check of how much drift may have occurred.

11. References

- 11.1. USEPA. 2004. pH Electrometric Measurement. Method 9040C (Rev. 3) Online at www.epa.gov/sw-846/pdfs/9040c.pdf. Accessed 3/7/2006.
- 11.2. Thermo Electron Corporation. 2006. Orion 301 PerpHecT® pH/Temperature Meter – Product Specifications. Online at <http://thermofinnigan.com/com/cda/product/detail/1,1055,10640,00.html>. Accessed 3/13/2006.
- 11.3. Hoskins, B. 2003. “Laboratory Procedures”. Recommended Methods of Manure Analysis (J. Peters, ed.). University of Wisconsin-Madison Cooperative Extension Publishing, pp. 12-13. Online at <http://learningstore.uwex.edu/Recommended-Methods-of-Manure-Analysis-P106C0.aspx>. Accessed 9/6/2007.
- 11.4. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 11.5. SOP M1. 2006. Manure Sampling. Standard Operating Procedure M1. Purdue Ag Air Quality Lab.

DETERMINING SOLIDS CONTENT OF MANURE SAMPLES
Standard Operating Procedure (SOP) M3

DETERMINING SOLIDS CONTENT OF MANURE SAMPLES

Standard Operating Procedure (SOP) M3

Prepared by

Alan L. Sutton, Brian Richert, Scott Radcliffe, Dan Kelly and Bill W. Bogan

Reviewed by

**Albert J. Heber, Harold M. Keener (Ohio State Univ.) and Jerome J. King
(Midwest Laboratories, Inc)**

Effective Date: December 28, 2007

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Health and Safety	3
4. Cautions	3
5. Interferences	4
6. Personnel Qualifications.....	4
7. Equipment and Supplies	5
8. Procedures	5
9. Data and Records Management	6
10. Quality Control and Quality Assurance	6
11. References	8

1. Scope and Applicability

- 1.1. Manure composition significantly affects its emissions of odor and individual chemical components. The solids-to-liquids ratio is an important property.
- 1.2. This method determines the total solids, volatile solids and fixed solids contents of a manure sample.
- 1.3. The method described in this SOP is taken from USEPA Method 1684 (2001 Draft version), and is therefore also applicable to soils, sediments, and both raw and processed sludges.
- 1.4. The stated Method Detection Limits (MDLs) specified for this method are 3 mg/L total solids, 7 mg/L volatile solids, and 7 mg/L fixed solids.

2. Summary of Method

Well-mixed sample aliquots, having a wet weight of between 25 and 50 g, are dried at 103°C to 105°C in order to drive off all of the moisture in the sample. This step allows for the determination of total solids. Following cooling, the total-solids portion of the sample is heated to 550°C, in a step that causes the volatile solids to be released. The sample is again cooled, and the remaining residue represents the fixed-solids portion.

3. Definitions

- | | |
|-------------------|---|
| 3.1. IPR | Initial precision & recovery |
| 3.2. MDL | Method detection limit |
| 3.3. OPR | Ongoing precision & recovery |
| 3.4. QA | Quality Assurance |
| 3.5. QC | Quality Control |
| 3.6. Sample batch | All samples which are analyzed at the same time, up to a maximum of twenty (20). If more than 20 samples are analyzed at once, they must be assigned into more than one batch for the purposes of including blanks and OPR standards. |

4. Health and Safety

- 4.1. Use gloves, tongs, and/or suitable sample holders when handling/moving hot sample vessels.

5. Cautions

- 5.1. When working with wet samples, weigh as quickly as possible after transferring to crucibles (Step 9.2.1), as these can lose water due to evaporation.
- 5.2. Unless extended drying times are required due to crusting (Section 6.3), do not exceed the drying times specified in this method. Overdrying can result in negative errors due to volatilization of certain salts (Section 6.5), volatilization of organic matter, or heat-

induced chemical decompositions, or to positive errors due to weight gain through sample oxidation.

- 5.3. Minimize the time that desiccators containing dried samples are open, as samples can adsorb moisture from the air. If strong desiccants are present in the sample (i.e. if weight gain occurs while samples are in the desiccator), use of a vacuum desiccator would be necessary. In all cases, do not remove samples from desiccators until immediately prior to weighing.

6. Interferences

- 6.1. Keep samples at or below 4°C after collection and during shipping (SOP M1), and prior to analysis, to minimize microbial degradation of sample solids. Analyze all samples within 7 days of collection. Frozen (archived) samples can be maintained indefinitely.
- 6.2. Samples must be kept homogeneous during the transfer of subsamples to drying vessels, as phase separation at this stage can be a source of considerable error.
 - 6.2.1. Use a magnetic stirrer (See Step 9.2.1.1).
 - 6.2.2. If the sample contains visible suspended solids, do not retrieve subsamples with a narrow-bore pipette. Use wide-bore pipettes, spoons or similar appropriate tools.
 - 6.2.3. If part of the whole sample adheres to the sample container walls despite magnetic stirring, a more intensive homogenization method must be used for mixing. In these cases, sample will be transferred (including scraping of the sample which is adhering to the vessel walls) into a Waring blender and homogenized in this way.
 - 6.2.4. Do not use a magnetic stirrer if magnetic particles are present in the sample.
- 6.3. If samples form a crust during the drying stage, this can inhibit further evaporation (either of water or volatile solids), and must be dealt with through special handling (either extended drying times or careful physical disruption (breakage) of the crust).
- 6.4. Following the initial drying step for total solids determination, some samples may retain small amounts of bound water, either as water of crystallization or interstitial water in crystals. Because the loss of this water is generally very slow, these samples may take longer to reach a constant weight.
- 6.5. Some samples may exhibit loss of CO₂ as bicarbonate is converted to carbonate. Loss of ammonium carbonate may also occur.
- 6.6. Samples with high levels of oil and grease may require particularly long drying times to reach a constant weight; it may therefore be difficult to obtain incontrovertible results for these samples.
- 6.7. Some dried samples may be highly hygroscopic, and contain stronger desiccants than are used in the desiccators themselves. Follow Section 5.3.
- 6.8. Use the same balance for all weighing steps involving a particular batch of samples.

7. Personnel Qualifications

- 7.1. Personnel should be trained in the use of the analytical instruments.
- 7.2. Each analyst must read and understand USEPA Method 1684 and this SOP before initiating these procedures, and must demonstrate the ability to generate acceptable data accuracy and precision as outlined in Section 11.2.

8. Equipment and Supplies

- 8.1. Drying oven
- 8.2. Muffle furnace
- 8.3. 15-100-mL crucibles or evaporating dishes made of porcelain or high-silica glass.
- 8.4. Watch glasses of suitable size to cover the crucibles/evaporating dishes
- 8.5. Analytical balance: Sartorius Model CP124S, Sartorius AG, Goettingen, Germany (or equivalent) (Capacity = 120 g; Accuracy = 0.0001 g).
 - 8.5.1. Reference masses for calibration: 2-mg, 1000-mg, and 50-g class "S" weights
- 8.6. Magnetic stirrer
- 8.7. Spatulas
- 8.8. Desiccator and desiccant (Drierite, Anhydrous Calcium Sulfate)

9. Procedures

- 9.1. Preparation
 - 9.1.1. If volatile solids are to be determined, heat crucibles/evaporating dishes and watch glasses to 550°C for 1 h.
 - 9.1.2. If only total solids are to be determined, heat crucibles/evaporating dishes and watch glasses to 103°C to 105°C for 1 h.
 - 9.1.3. Cool and store all glassware in a desiccator.
 - 9.1.4. Prior to use, weigh empty clean crucible and watch glass, and record value (to the nearest 0.01 g) of the two combined.
- 9.2. Sample handling
 - 9.2.1. Obtaining subsamples
 - 9.2.1.1. Fluid (i.e. free-flowing) samples: Stir to homogenize (either by magnetic stirrer or using a spatula). Taking care that the sample remains homogeneous (i.e. that no phase separation occurs), transfer a 25- to 50-g aliquot to a crucible/dish using a pipette, spoon, or other appropriate tool. Spread evenly over the crucible. Replace watch glass and re-weigh; record weight to the nearest 0.01 g.
 - 9.2.1.2. Wet solid samples: Stir with a spatula to homogenize. Transfer a 25- to 50-g aliquot to a crucible/dish using a spatula, spoon, or other appropriate tool. Spread evenly over the crucible. Replace watch glass and re-weigh; record weight to the nearest 0.01 g.
 - 9.2.1.3. Dry solid samples: Pulverize the entire sample by hand (using rubber gloves) on a clean surface. Mix, and transfer a 25- to 50-g aliquot to a crucible/dish using a spatula, spoon, or other appropriate tool. Spread evenly over the crucible. Replace watch glass and re-weigh; record weight to the nearest 0.01 g.
 - 9.2.2. Include one blank (Section 11.8) and one Ongoing Precision and Recovery sample (Section 11.3) with each batch of samples.
 - 9.2.3. Put in oven to dry for 12 h at 103°C to 105°C.
 - 9.2.4. Remove from oven with tongs and place in the desiccator to cool to room temperature. Do not remove from desiccator until immediately prior to weighing. See Step 5.3 if working with highly hygroscopic samples.
 - 9.2.5. Weigh cooled crucible/watch glass/sample, and record value to the nearest 0.01 g.

- 9.2.6. Place crucible in the (cool) muffle furnace and raise temperature to 550° C and hold for 2 h.
- 9.2.7. Remove crucible from the muffle furnace with tongs and place in the desiccator to cool to room temperature.
- 9.2.8. Weigh and record value of crucible/watch glass/ash residue to the nearest 0.01 g.
- 9.2.9. Return to muffle furnace for 30 minutes, cool and re-weigh. Repeat as necessary until weight change is less than 4% or 50 mg (whichever is less).
- 9.2.10. Record final value of crucible/watch glass/ash residue to the nearest 0.01 g.
- 9.3. Calculations
 - 9.3.1. Wet sample weight:
Crucible/watch glass/wet sample (Step 9.2.1) – Crucible/watch glass (Step 9.1.4)
 - 9.3.2. Dry sample weight:
Crucible/watch glass/dry sample (Step 9.2.7) – Crucible/watch glass (Step 9.1.4)
 - 9.3.3. Ash weight:
Crucible/watch glass/ashed sample (Step 9.2.12) – Crucible/watch glass (Step 9.1.4)
 - 9.3.4. % Total Solids = ((Dry sample weight)/(Wet sample weight)) * 100
 - 9.3.5. % Fixed Solids = ((Ash weight)/(Dry sample weight)) * 100
 - 9.3.6. % Volatile Solids = 100% - % Fixed Solids

10. Data and Records Management

- 10.1. Maintain all laboratory records in an electronic spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
- 10.2. Document all data and information on data sheets with permanent ink, or in electronic field notes.

11. Quality Control and Quality Assurance

- 11.1. Quality Control (QC) Solutions
 - 11.1.1. One of the following solutions will be employed for QC checks of the procedures described in this SOP:
 - 11.1.1.1. NaCl-KHP QC solution
 - 11.1.1.1.1. Dissolve 0.10 g NaCl in reagent-grade (de-ionized) water.
 - 11.1.1.1.2. Add 0.10 g potassium hydrogen phthalate (KHP), and mix to dissolve. Warm and stir if necessary to dissolve the KHP.
 - 11.1.1.1.3. Dilute with de-ionized water to 1.0 L in a volumetric flask.
 - 11.1.1.1.4. Store at 4°C
 - 11.1.1.1.5. This solution contains 200 mg/L of total solids, 81 mg/L of volatile solids, and 119 mg/L fixed solids.
 - 11.1.1.2. Commercially-obtained QC solution: Wibby Environmental Volatile Solids QC Standard (Catalog # QC-VSOL-WP), Wibby Environmental, Golden, CO (or equivalent).

11.2. Analyst Proficiency Demonstration

11.2.1. Initial Precision and Recovery (IPR): Analyst must demonstrate ability to generate acceptable precision and accuracy. Prepare at least four (4) replicate (50-mL) aliquots of a 12-fold dilution of the QC solution. Process all replicates through each step of the analytical method, and perform all calculations. Using the results of the four or more replicates, calculate the average and standard deviation for the concentrations of total, volatile and fixed solids. Compare to the following table:

Analyte	Expected value	Acceptable range	Acceptable S.D.
Total solids (mg/L)	16.7	14.2 – 18.4	10%
Volatile solids (mg/L)	9.92	7.44 – 10.91	30%
Fixed solids (mg/L)	6.75	5.06 – 7.42	20%

11.2.1.1. If averages and standard deviations do not fall within these acceptable ranges, analyst must identify his/her source(s) of error and repeat the IPR procedure.

11.3. Ongoing Precision and Recovery (OPR)

11.3.1. Prepare an OPR solution identical to the IPR solution described in Step 11.2.1 (i.e. a 12-fold dilution of the QC solution).

11.3.2. Include one (1) OPR sample (25-50 mL) with each sample batch, and subject it to all steps of the analytical method.

11.3.3. Concentrations determined from the OPR sample must fall within the following ranges (in mg/L):

- Total solids: 13.4 – 18.4
- Volatile solids: 6.94 – 10.91
- Fixed solids: 4.90 – 7.42

11.3.4. If recovery of any analyte falls outside the acceptable range for a given OPR sample, repeat analysis of that sample batch after appropriate troubleshooting and correction of the problem.

11.4. QC Charts

11.4.1. Track all IPR and OPR results to keep a graphical representation of continued laboratory performance.

11.5. Method Modifications & Re-validation

11.5.1. Each time any modification is made to this method, the analyst will verify that the modified method meets or exceeds the Method Detection Limit and Initial Precision and Recovery achieved by the same analyst using the original (unmodified) method.

11.5.2. Verify MDLs by diluting either the NaCl-KHP QC solution (Section 11.1.1.1) or the commercial standard (Section 11.1.1.2) to the extent(s) necessary to achieve the solids contents listed in Section 1.4. Analyze each of these diluted solutions by the modified method to verify that the analyte can be detected at that level.

11.5.3. The laboratory will maintain records of any modifications made to the method. At minimum, these records must include the following:

- 11.5.3.1. Names, titles, and contact information for analyst who initiated the modifications and for the QA officer who witnessed and approved the modifications.
- 11.5.3.2. Narrative stating reason(s) for the modification

11.5.3.3. Results from all QC tests comparing the modified method's performance to that of the original method:

- Initial Precision and Recovery
- Accuracy
- Blanks
- Ongoing Precision and Recovery

11.6. Data Integrity

11.6.1. A second person will be present to verify that analyses were conducted properly and provide a signature at the end of the data set run and/or the end of the working day.

11.7. Replication

11.7.1. Run at least 10% (one in ten) of the samples in each batch in duplicate, with at least one duplicate per batch. Initial masses (i.e. wet weights) of the duplicates will not differ by more than 10%. Replicates must agree to within 10% of their average (% total solids); if not, rerun this batch.

11.8. Blanks

11.8.1. One blank (25-50 mL of deionized water) must be included with each sample batch, and be subjected to all steps of the analytical method.

11.8.2. If material is detected in the blank at or above the MDLs stated in Section 1.4, stop analysis until the source of contamination is identified and eliminated (i.e. a new blank shows no evidence of contamination).

11.9. Instrument Calibration and Standardization

11.9.1. Analytical balance

11.9.1.1. Full calibration (annually at a minimum).

11.9.1.1.1. Calibrate at 2 mg and 1000 mg using class "S" weights.

11.9.1.1.2. Instrument readings must be within $\pm 10\%$ at 2 mg (i.e. ± 0.2 mg) and $\pm 0.5\%$ at 1000 mg (i.e. ± 5 mg). If these are not achieved, re-calibrate the balance.

11.9.1.2. Calibration check (daily before any samples are weighed)

11.9.1.2.1. Place a 50-g weight and the 2-mg weight on the balance together. Verify that the balance reads 50.002 g (± 0.0002 g).

12. References

- 12.1. USEPA. 2001. Method 1684 – Total, Fixed, and Volatile Solids in Water, Solids, and Biosolids. EPA-821/R-01-015 (CD). Office of Water, Office of Science and Technology. Washington, DC.
- 12.2. Hoskins, B. 2003. "Laboratory Procedures". Recommended Methods of Manure Analysis (J. Peters, ed.). University of Wisconsin-Madison Cooperative Extension Publishing, pp. 12-13. Online at <http://learningstore.uwex.edu/Recommended-Methods-of-Manure-Analysis-P106C0.aspx>. Accessed 9/6/2007.
- 12.3. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 12.4. SOP M1. 2006. Manure Sampling. Standard Operating Procedure M1. Purdue Ag Air Quality Lab.

**DETERMINING TOTAL (KJELDAHL) NITROGEN CONTENT
OF MANURE SAMPLES
Standard Operating Procedure (SOP) M4**

**DETERMINING TOTAL (KJELDAHL) NITROGEN CONTENT
OF MANURE SAMPLES
Standard Operating Procedure (SOP) M4**

Prepared by

Alan L. Sutton, Brian Richert, Scott Radcliffe, Dan Kelly and Bill W. Bogan

Reviewed by

Albert J. Heber and Jerome J. King (Midwest Laboratories, Inc)

Effective Date: December 28, 2007

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability.....	3
2. Summary of Method.....	3
3. Definitions.....	3
4. Health and Safety.....	4
5. Cautions.....	4
6. Interferences.....	4
7. Personnel Qualifications.....	5
8. Equipment and Supplies.....	5
9. Procedures.....	7
10. Data and Records Management.....	8
11. Quality Control and Quality Assurance.....	8
12. References.....	12

1. Scope and Applicability

- 1.1. Manure composition can significantly affect its emissions, both in terms of general odor and individual chemical components. The total nitrogen content is an important manure property that affects emission of ammonia and other nitrogen-containing compounds. Thus, this parameter is important for both barn and open-source systems.
- 1.2. This method determines the total Kjeldahl nitrogen (TKN) content of a manure sample, Kjeldahl nitrogen being the sum of ammoniacal nitrogen and organic nitrogen.
- 1.3. The digestion method described in this SOP is taken from USEPA Method 1687 (2001 draft version), and is therefore also applicable to wastewaters, drinking water or groundwater, and industrial and other wastes.
- 1.4. The distillation and titrimetric procedures are described in USEPA Method 351.3.
 - 1.4.1. This method has a working range of ca. 0.5-10 mg N/g manure (wet weight basis).
 - 1.4.2. The stated detection limit for the method (Reference 12.4) is 0.01% (0.1 mg/g) N.

2. Summary of Method

Organic nitrogen in a manure sample is first converted to ammonia by metal-catalyzed acid digestion (with the exceptions listed in Section 6.7). The ammonia in the digested sample (the sum of nitrogen that was originally present in the ammoniacal form, plus that which is liberated from organic compounds) is then distilled away from the rest of the sample, at which point it is captured in a dilute sulfuric acid/boric acid solution containing bromocresol green/methyl red indicator. The ammonia concentration of the distillate is then determined by titration with sulfuric acid. Cupric sulfate/titanium dioxide ($\text{CuSO}_4/\text{TiO}_2$) is used as the sole catalyst in the digestion process. This avoids the toxicity and waste-disposal problems posed with mercury- and/or selenium-containing compounds. A QA/QC section is provided to discuss the calibration and periodic testing of the analytical method to ensure reliable data.

3. Definitions

- | | | |
|-------|--------------|--|
| 3.1. | COD | Chemical oxygen demand |
| 3.2. | CV | Calibration verification |
| 3.3. | IPR | Initial precision and recovery |
| 3.4. | MDL | Method detection limit |
| 3.5. | MgO | Magnesium oxide |
| 3.6. | MS | Matrix spike |
| 3.7. | NaOH | Sodium hydroxide |
| 3.8. | OPR | Ongoing precision and recovery |
| 3.9. | QA/QC | Quality assurance/quality control |
| 3.10. | Sample batch | A group of samples analyzed at the same time, up to a maximum of twenty (20). If more than 20 samples are analyzed at a time, divide them into multiple batches for the purpose of including blanks and CV and PR standards. |
| 3.11. | SOP | Standard operating procedure |
| 3.12. | TKN | Total Kjeldahl nitrogen |

4. Health and Safety

- 4.1. Use gloves, tongs, and/or suitable sample holders when handling hot sample tubes.
- 4.2. Treat each chemical compound as a potential health hazard, and reduce exposure to chemicals to the lowest possible level.
 - 4.2.1. Wear protective clothing (gloves, lab coats, protective eyewear) when working with chemicals.
 - 4.2.2. Conduct all sample digestions in properly-ventilated fume hoods. Use a manifold to remove acid fumes.
 - 4.2.3. Keep analytical balances and other work areas clean; deal promptly with spills.
 - 4.2.4. When using concentrated acids and bases, keep a fully-stocked spill kit (including suitable neutralizers and absorbents) in the work area.
- 4.3. Maintain a current awareness file of Occupational Safety and Health Administration (OSHA) regulations pertaining to the chemicals specified in this method.
- 4.4. Maintain a reference file of material safety data sheets, which will be available to all personnel involved in these analyses.
- 4.5. Sulfuric acid digest is classified as a hazardous waste. Dispose of accordingly.

5. Cautions

- 5.1. Considerable heat is generated when dissolving sodium hydroxide (NaOH) in water (Step 8.7.2), or by mixing acids with water. Cool the container in an ice bath when preparing NaOH solutions or when mixing acid and water.
- 5.2. Excessive foaming during distillation can cause foam to pass into the condenser, which can contaminate the receiving flask and condenser with alkaline salts. Treat heavily-foaming samples with an anti-foaming agent.

6. Interferences

- 6.1. Keep samples at or below 4°C after collection and during shipping (SOP M1), and prior to analysis, to minimize microbial activity. Analyze all samples within 7 days of collection. Frozen (archived) samples can be maintained indefinitely.
- 6.2. Use only ammonia-free water, which can be prepared by passing reagent water through an ion-exchange column that is packed with a mixture of both strongly acidic cation- and strongly basic anion-exchange resins. Regenerate this column periodically according to the manufacturer's instructions.
- 6.3. Ammonia gas in the lab can deposit into the sample. Minimize sample exposure to air.
- 6.4. Nitrate in large quantities (>10 mg/L) will oxidize ammonia produced by the digestion and can introduce a substantial negative error. Although this interference cannot be prevented, its effect can be predicted and accounted for on the basis of preliminary nitrate determination of the sample. Whenever high nitrate levels are a potential issue, nitrate analyses of the sample should be conducted first.

Inorganic salts or solids will increase digestion temperature; however, conducting digestion at temperatures in excess of 400°C will cause pyrolytic loss of nitrogen. If high levels of salts and/or solids are suspected, adding extra H₂SO₄ can partially circumvent this problem. Approximately 1 mL H₂SO₄ per g of salt/solid will preserve the proper ratio, and allow digestion to proceed at the target temperature. Monitoring of digestion temperature will indicate if problems are occurring.

- 6.5. Large amounts of organic matter can consume the acid in the digestion reagent, causing the digestion temperature to rise above 400°C. Adding 10 mL H₂SO₄ per 3000 mg COD will prevent this. Monitor digestion temperature and pH if this correction is used.
- 6.6. Nitrogen occurring in certain organic compounds will not be measured by this method. These compounds include those in which the nitrogen occurs in the following forms:

- azide
- azine
- azo
- hydrazone
- nitrate
- nitrite
- nitrile
- nitro
- nitroso
- oxime
- semicarbazone

- 6.6.1. Levels of these compounds in manure are expected to be negligible.

7. Personnel Qualifications

- 7.1. Personnel should be trained in the use of the equipment before initiating the procedure.
- 7.2. Each analyst must read and understand this SOP, as well as USEPA Methods 1687 and 351.3, before initiating this procedure, and must show the ability to generate acceptable data accuracy and precision (Section 11.3).

8. Equipment and Supplies

- 8.1. Copies of USEPA Methods 1687 and 351.3
- 8.2. Tecator Instrument Model 2020 digester block and aspirator for sample digestion
- 8.3. Foss Kjeltec Model 2300 automated distillation and titration analyzer (FOSS Analytical, Hilleroed, Denmark), or equivalent
- 8.4. Flasks: 100-mL Kjeldahl and 50-mL Erlenmeyer flasks
- 8.5. Magnetic stirrer and Teflon-coated stir bars
- 8.6. Spatulas
- 8.7. Analytical balance (Capacity = 410 g; Accuracy = 0.001 g)
- 8.7.1. Reference weights for calibration: 2-mg, 1000-mg, and 50-g class “S” weights
- 8.8. Reagents
- 8.8.1. Sulfuric acid: Concentrated, 0.020N & 0.25N H₂SO₄, certified (Midland Scientific, Omaha, NE)
- 8.8.2. Sodium hydroxide: 40% NaOH solution, certified (Midland Scientific, Omaha, NE)

- 8.8.3. Digestion reagent: TT-43 Pro Pac Kjeldahl digestion tablets (Alfie packers, Inc, Omaha, NE)
- 8.8.4. Boric acid indicator
 - 8.8.4.1. Add 0.10 g bromocresol green, 0.10 g methyl red, 100 g boric acid, and 20 mL of 0.10N NaOH to a 2-L flask that is half-full of NH₃-free water and dissolve all reagents
 - 8.8.4.2. Add NH₃-free water to the 2-L mark on the flask.
 - 8.8.4.3. Quantitatively transfer contents of the flask to the receiving solution reservoir below the Foss Kjeltech Model 2300.
 - 8.8.4.4. Add 8 moreL of NH₃-free water to the reservoir, for a total of 10 L of solution.
- 8.9. Standards
 - 8.9.1. Ammonia standards
 - 8.9.1.1. Ammonia stock solution (1000 mg/L NH₃-N): Dissolve 0.382 g of anhydrous ammonium chloride (NH₄Cl), which should be dried at 105°C immediately prior to use, in NH₃-free water and dilute to 100 mL in a volumetric flask with NH₃-free water.
 - 8.9.1.2. Ammonia working standard (10 mg/L NH₃-N): Dilute 10 mL of ammonia stock solution to 1 L in a volumetric flask with NH₃-free water.
 - 8.9.2. Nicotinic acid standard (Contains 100 mg/L organic N): Dissolve 21.637 g nicotinic acid in ~ 150 mL NH₃-free water. Dilute to 200 mL with NH₃-free water.
 - 8.9.3. Calibration standards
 - 8.9.3.1. Prepare calibration standards by spiking 5-g aliquots of blank sand or diatomaceous earth (Section 8.10) directly with nicotinic acid standard solution (Section 8.9.2).
 - 8.9.3.2. Prepare a minimum of five calibration standards, at five different concentrations to produce a calibration curve with at least five points.
 - 8.9.3.3. At least one of the calibration standards should correspond to a sample concentration at or below that necessary to meet the data quality objectives of the project. The QAPP for a particular project will state the specific concentration needed for this standard.
 - 8.9.3.4. The remaining standards should correspond to the concentration range found in actual samples, without exceeding the titration-based system's working range.
 - 8.9.4. Calibration Verification (CV) standard
 - 8.9.4.1. Prepare the CV standard such that its N concentration approximates the midpoint of the calibration curve.
 - 8.9.4.2. The source used to prepare the CV standard should be different from that used to prepare the calibration standards. Use either a different bottle of nicotinic acid, or a different compound altogether.
 - 8.9.4.3. If a different ammonium compound is used for the CV stock, adjust the amount weighed according to the ratio of N weight to the molecular weight. Nicotinic acid has one atom of N per molecule and a formula weight of 123.11; it is, therefore 11.37% N by weight (14/123.11). If, for example, nicotinamide (2 N per molecule, formula weight 122.12, 22.93% N) is used as the CV standard, and X mg of nicotinic acid was needed to yield the desired N concentration, then (11.37/22.93) X, or 0.496 X g of nicotinamide would be needed.

- 8.9.5. Quality control sample (QCS): Standard Reference Material 1570a (Spinach Leaf). National Institute of Standards & Technology, Gaithersburg, MD.
- 8.10. Blank sand or diatomaceous earth
 - 8.10.1. Bake 500 g diatomaceous earth or clean sand at 400°C for 8 h. Cool and store in a glass container with a sealing lid.

9. Procedures

9.1. Sample Preparation

- 9.1.1. In a fume hood, preheat the digestion block of the Model 2020 to 380°C.
- 9.1.2. Obtain percent total solids of each sample to be analyzed, according to SOP M3.
- 9.1.3. Label digestion tubes as necessary to accommodate all samples, plus the various QA standards (Duplicates, Precision and Recovery Samples, Calibration Verification Standards, Quality Control Standards, Matrix Spikes, and/or Blanks) as required (See Sections 11.4 through 11.9 for the frequency that these must be included).
- 9.1.4. Bring samples to room temperature. Thoroughly homogenize the sample. Transfer 50 mL of sample into a digestion tube, and obtain and record the sample weight.
- 9.1.5. Add Kjeldahl digestion tablets (Section 8.8.3) and 20 mL of concentrated H₂SO₄, and place tube in the preheated digestion block.

9.2. Digestion

- 9.2.1. Immediately set up the exhaust system, and place the exhaust manifold so that it covers the mouth of each digestion tube. Turn water to vacuum on low.
- 9.2.2. Digest for 2 h.
 - 9.2.2.1. Be sure that the manifold and hood fan are operating properly to remove the fumes released in the digestion process.
- 9.2.3. Remove tubes from digestion block with tongs and transfer to cooling tray to cool, leaving the exhaust manifold in place for a minimum of 30 min after the end of digestion. After 30 min, turn off water to vacuum, and remove exhaust manifold.
- 9.2.4. Add NH₃-free water to each tube to the level of the top horizontal support on the cooling rack (~50 mL). Place samples in the cooler and cool to <20 °C.

9.3. Distillation

- 9.3.1. Place samples on the Model 2300 and select “Kjeldahl 5”. This selection ensures that the instrument will dispense a sufficient amount of NaOH to the sample.
 - 9.3.1.1. Use 0.020 N H₂SO₄ for low-N samples, and 0.25 N for all others. Most samples associated with livestock manure, feed, and bedding are assumed to require 0.25 N H₂SO₄.

9.4. Calculations

- 9.4.1. Calculate TKN (in units of mg/g) in the sample as follows, correcting the amount of titrant (0.25N H₂SO₄) added to each sample by subtracting that needed to titrate a blank to endpoint:

$$\text{TKN} = \frac{\text{Titrant}_{\text{sample}} (\text{mL}) - \text{Titrant}_{\text{blank}} (\text{mL})}{\text{Sample wt (g)}} \times 3,500^*$$

*If 0.25N H₂SO₄ is used in the distillation/titration step. If 0.02 N acid is used, replace 3,500 with 280.

10. Data and Records Management

- 10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
- 10.2. Manage all data according to SOP B5.
- 10.3. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes. Overstrike errors in writing with single lines.
- 10.4. Initial and date all corrections.

11. Quality Control and Quality Assurance

11.1. Reagent Purity

11.1.1. Use only NH₃-free water in this method. See Step 6.1 for the methodology used to remove ammonia from water for use in these procedures.

11.1.2. All other reagents will be ACS Reagent Grade or better.

11.2. Instrument Calibration and Standardization

11.2.1. Calibrate analytical balance annually (at a minimum).

11.2.1.1. Calibrate at 2 mg and 1000 mg using class "S" weights.

11.2.1.2. Instrument calibration must be within $\pm 10\%$ at 2 mg (i.e. ± 0.2 mg) and $\pm 0.5\%$ at 1000 mg (i.e. ± 5 mg). If these limits are not achieved, the balance must be re-calibrated.

11.2.1.3. Place a 50-g weight and the 2-mg weight on the balance together. Verify that the balance reads 50.002 g (± 0.0002 g).

11.3. Initial Demonstration of Analyst & Laboratory Proficiency

11.3.1. The following steps are to be followed by every new analyst performing this method, in order to assure the quality of his/her technique. No analyst may perform analyses of actual samples until he/she has passed all of the following:

11.3.1.1. Determine Method Detection Limit (MDL)

11.3.1.1.1. Prepare a group of at least seven (7) MDL determination samples by mixing 5-g aliquots of blank sand (or diatomaceous earth) (Section 8.10) with 10 mL NH₃-free water, and spiking each with nicotinic acid standard (Section 8.9.2) such that the organic N concentration of each MDL sample is one to five times the estimated detection limit (Section 1.4.2).

11.3.1.1.2. Using the seven replicates; process each through every step of the method. Calculate the N concentrations (Section 9.4.1) for each aliquot, and compare with the expected concentrations.

11.3.1.1.3. If, on average, the calculated concentrations are within 20% of the expected concentrations, analyst and system performance are acceptable .

11.3.1.2. Initial Precision and Recovery (IPR)

11.3.1.2.1. Prepare a group of at least four (4) IPR samples by mixing 5-g aliquots of blank sand (or diatomaceous earth) with 10 mL NH₃-free water, and spiking the slurries with nicotinic acid standard so that the organic N concentration of each PR sample is on the order of 1%. Process each sample through every step of the method. Perform the TKN calculations in Section 9.4.1, and determine

- the percent recovery by comparing these to the known input amount.
- 11.3.1.2.2. Using the results of the set of four analyses, compute the average percent recovery (X) and the standard deviation (s) of the percent recovery for TKN.
 - 11.3.1.2.3. The required performance specifications are $110\% \geq X \geq 90\%$, and $s \leq 5\%$.
 - 11.3.1.2.4. If X and s meet the acceptance criteria, analyst and system performance are acceptable..
 - 11.3.1.2.5. If s exceeds the precision limit (5%), and/or X falls outside the range for recovery, analyst and/or system performance is unacceptable. In this event, correct the problem, and repeat the test.
- 11.3.1.3. Blank
- 11.3.1.3.1. Include a minimum of one blank (Section 11.9) to verify that nitrogen is not detected in the blank at a concentration greater than the aqueous or solid MDL (Section 11.4.1.1).
- 11.3.1.4. Linearity of calibration curve
- 11.3.1.4.1. Analyst will prepare a five-point calibration curve (Section 8.9.3) to verify his/her ability to generate linear results over the concentration range of interest. If the linear correlation coefficient (r^2) of the calibration curve is ≥ 0.96 , analyst and system performance are acceptable.
- 11.3.1.5. CV Standard
- 11.3.1.5.1. Include a minimum of one CV Standard (Section 8.9.4) to verify that the analyst can meet the acceptance criteria outlined in Section 11.6.1.1.
- 11.4. Ongoing Precision and Recovery (OPR)
- 11.4.1. MDL
- 11.4.1.1. Re-determine/verify MDLs annually.
 - 11.4.1.2. Re-determine/verify MDLs whenever a modification is made to the method or analytical system.
- 11.4.2. Precision and Recovery (PR) samples
- 11.4.2.1. Each batch of samples (Section 3.10) must contain a minimum of one PR sample. Run solid samples with solid PR samples, and slurry samples with slurry PR samples.
 - 11.4.2.1.1. Prepare a solid PR sample by spiking 5 g of blank sand (or diatomaceous earth) with nicotinic acid standard such that the organic N concentration of the PR sample is approximately five times the MDL (Section 1.4.2).
 - 11.4.2.1.2. Prepare a slurry PR sample by mixing 5 g of blank sand (or diatomaceous earth) with 10 mL NH_3 -free water, and spiking the slurry with nicotinic acid standard such that the organic N concentration of the PR sample is approximately five times the MDL (Section 1.4.2).
 - 11.4.2.2. Carry the spiked PR sample through the entire analytical process, and calculate percent recovery.
 - 11.4.2.2.1. If recovery falls within 90 to 110%, the system performance is acceptable.
 - 11.4.2.2.2. If recovery falls outside 90% to 110%, system performance is unacceptable, and the problem must be identified and resolved prior to further analyses.
 - 11.4.2.3. All samples must be associated with an acceptable PR standard before their results may be reported.
- 11.5. Calibration curves

- 11.5.1. Generate a new calibration curve (containing a minimum of five points as described in Section 8.9.3) whenever a new standard is obtained.
- 11.6. Calibration Verification (CV) standards
- 11.6.1. A Calibration Verification Standard (solid or slurry, as appropriate) must be run once per sample batch (every twenty analyses – Section 3.10).
- 11.6.1.1. Acceptance criteria for the CV Standard is $100\% \pm 5\%$.
- 11.6.1.2. If the CV does not meet acceptance criteria, the problem must be identified and corrected, including possible recalibration of the system if the problem cannot be corrected by other means.
- 11.6.1.3. All samples must be associated with an acceptable CV Standard before their results may be reported.
- 11.7. Quality Control standards
- 11.7.1. In accordance with Method 1687, analyze a QC standard with each day's distillations, or every 12 h, whichever is more frequent.
- 11.8. Matrix Spikes
- 11.8.1. To assess the performance of the method on a given sample matrix, spike (in duplicate) a minimum of 5% (1 sample in 20) of the samples from a given site.
- 11.8.2. Ensure that the concentration of nitrogen spiked into each MS sample is 1-5 times the background (i.e. existing) concentration expected for that particular sample.
- 11.8.2.1. The spiking level may be established *a priori* based on existing information (either analyst's previous experience or existing literature values) for N content of manures from the particular species.
- 11.8.3. The concentration added should be expressed as mg/kg, and should be calculated for a 1-g aliquot by multiplying the N concentration (mg/L) added (as nicotinic acid) by the conversion factor $100 \text{ (mg/L} \times 0.1\text{L}/0.001\text{kg} = 100)$.
- 11.8.4. Do not use blanks for MS analysis.
- 11.8.5. Assessing spike recovery
- 11.8.5.1. Calculate the percent recovery of TKN in the duplicate matrix spike samples (corrected for the background concentration measured in the corresponding unspiked sample which was used as the basis for the MS samples), as well as the relative percent difference (RPD) between the two values.
- 11.8.5.1.1. Percent recovery is calculated as follows:

$$\text{Percent Recovery} = \frac{(C_s - C_b)}{S} \times 100$$

Where:

C_s = Sample N concentration determined after spiking

C_b = Sample N concentration determined before spiking

S = Known concentration of the spike

- 11.8.5.1.2. RPD is calculated as follows:

$$\text{RPD} = \frac{(|D_1 - D_2|)}{D_1 + D_2} \times 100$$

Where:

D_1 = TKN concentration of MS sample #1

D_2 = TKN concentration of MS sample #2

- 11.8.5.2. Percent recoveries for the matrix spikes should be within three (3) standard deviations above or below the mean, with $\text{RPD} \leq 20\%$.
- 11.8.5.3. If either the percent recovery or the RPD of TKN in the MS samples falls outside the designated range, while the percent recovery in the PR sample is within the acceptable range, the problem encountered with the MS sample is judged to be matrix-related instead of method-related.
- 11.9. Blanks
- 11.9.1. Run a minimum of one blank with each batch of samples (Section 3.10).
- 11.9.2. Run aqueous (slurry) samples with an aqueous blank (10 mL NH_3 -free water), and solid samples with a solid blank (a 5-g aliquot of blank sand).
- 11.9.3. If material is detected in the aqueous or solid blank at a concentration greater than the MDL (Section 1.4.2), halt analysis of samples until the source of contamination is eliminated and a new blank shows no contamination.
- 11.9.4. All samples must be associated with an uncontaminated laboratory blank before their results may be reported.
- 11.10. QC Charts
- 11.10.1. Track all IPR, OPR, Calibration Verification, QC Standard, MS and Blank results to provide graphical representations of continued laboratory performance.
- 11.11. Method modifications & re-validation
- 11.11.1. If any modification is made to this method, verify that the modified method meets or exceeds the Method Detection Limit (11.3.1.1) and Initial Precision and Recovery (11.3.1.2) achieved by the same analyst using the original (i.e. unmodified) method.
- 11.11.2. Maintain records of any modifications made to the method. At minimum, these records must include the following:
- 11.11.2.1. Names, titles, and contact information for analyst who initiated the modifications and for the QA Officer who witnessed and approved them
- 11.11.2.2. Narrative stating reason(s) for the modification
- 11.11.2.3. Results from all QC tests comparing the modified method's performance to that of the original method:
- Initial Precision and Recovery
 - Ongoing Precision and Recovery
 - Results with CV, QC Standard, MS samples and Blanks
- 11.12. Duplication
- 11.12.1. Run a minimum of 10% of samples (1 in 10) in duplicate, with a minimum of one duplicate per batch. Replicates must agree to within 10% of their average TKN content; if not, the batch of samples must be re-run.

12. References

- 12.1. Bremner, J.M. and D.R. Keeney. 1965. Steam distillation methods for determination of ammonium, nitrate and nitrite. *Analytica Chimica Acta*. 32:485-495.
- 12.2. USEPA Method 351.3. 1978. Nitrogen, Kjeldahl (Colorimetric, Titrimetric, Potentiometric). Online at http://www.umass.edu/tei/mwwp/acrobat/epa351_3Norg.pdf. Accessed 8/6/2007.
- 12.3. USEPA Method 1687. 2001. Total Kjeldahl Nitrogen in Water and Biosolids by Automated Colorimetry with Preliminary Distillation/Digestion (Draft). EPA-821/R-01-004 (CD). USEPA Office of Water, Office of Science and Technology. Washington, DC.
- 12.4. Watson, M., A. Wolf, and N. Wolf. 2003. "Total Nitrogen". Recommended Methods of Manure Analysis (J. Peters, ed.). University of Wisconsin-Madison Cooperative Extension Publishing, pp. 18-24. Online at <http://learningstore.uwex.edu/Recommended-Methods-of-Manure-Analysis-P106C0.aspx>. Accessed 9/6/2007.
- 12.5. Hoskins, B. 2003. "Laboratory Procedures". Recommended Methods of Manure Analysis (J. Peters, ed.). University of Wisconsin-Madison Cooperative Extension Publishing, pp. 12-13. Online at <http://learningstore.uwex.edu/Recommended-Methods-of-Manure-Analysis-P106C0.aspx>. Accessed 9/6/2007.
- 12.6. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 12.7. SOP M3. 2006. Determining Solids Content of Manure Samples. Standard Operating Procedure M3. Purdue Ag Air Quality Lab.
- 12.8. EN SOP 65e. 2004. Total Kjeldahl Nitrogen (TKN). Standard Operating Procedure EN65e. Midwest Laboratories, Inc, Omaha, NE.

DETERMINING AMMONIA CONTENT OF MANURE SAMPLES
Standard Operating Procedure (SOP) M5

DETERMINING AMMONIA CONTENT OF MANURE SAMPLES

Standard Operating Procedure (SOP) M5

Prepared by

Alan L. Sutton, Brian Richert, Scott Radcliffe, Dan Kelly and Bill W. Bogan

Reviewed by

Albert J. Heber and Jerome J. King (Midwest Laboratories, Inc)

Effective Date: December 28, 2007

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability.....	3
2. Summary of Method.....	3
3. Definitions.....	3
4. Health and Safety.....	3
5. Cautions.....	4
6. Interferences.....	4
7. Personnel Qualifications.....	4
8. Equipment and Supplies.....	5
9. Procedures.....	6
10. Data and Records Management.....	7
11. Quality Control and Quality Assurance.....	7
12. References.....	11

1. Scope and Applicability

- 1.1. The composition of manure can significantly affect its emissions, both in terms of general odor and individual chemical components. The ammonia content of a manure is generally a good indicator of readily available nitrogen, and therefore affects emission of both ammonia and other nitrogen-containing compounds.
- 1.2. This method determines a manure sample's total ammoniacal nitrogen (NH₃-N) content.
- 1.3. The distillation and titrimetric analyses are described in USEPA Method 350.2 and elsewhere (Bremner and Keeney 1965).
 - 1.3.1. This method has a working range of approximately 0.5-10 mg NH₃ per g of manure (wet weight basis), and a minimum detection limit of approximately 0.1 mg/g.
 - 1.3.2. The digestion and distillation method described in this SOP has been scaled down to "micro-Kjeldahl" scale in accordance with Reference 12.2.

2. Summary of Method

The ammonia in a manure sample is distilled away from the rest of the sample, at which point it is captured in a dilute boric acid solution which contains a bromocresol green methyl red indicator. The ammonia concentration of the distillate is then determined by titration with a known concentration of sulfuric acid. A quality assurance/quality control (QA/QC) section is provided to discuss the calibration and periodic testing of the analytical method to ensure that reliable data are collected.

3. Definitions

3.1. CV	Calibration verification
3.2. IPR	Initial precision and recovery
3.3. MDL	Method detection limit
3.4. MgO	Magnesium oxide
3.5. MS	Matrix spike
3.6. NaOH	Sodium hydroxide
3.7. OPR	Ongoing precision and recovery
3.8. PR	Precision and recovery
3.9. QA/QC	Quality assurance/quality control
3.10. RPD	Relative percent difference
3.11. Sample batch	A group of sample analyzed at the same time, up to a maximum of twenty (20). If more than 20 samples are analyzed at a time, they must be divided into multiple batches for including blanks and QA standards.
3.12. SOP	Standard operating procedure

4. Health and Safety

- 4.1. Treat each chemical compound as a potential health hazard, and reduce exposure to chemicals to the lowest possible level.

- 4.1.1. Wear protective clothing (gloves, lab coats, protective eyewear) when working with chemicals.
- 4.1.2. Conduct all operations involving solvents in properly-ventilated fume hoods.
- 4.1.3. Keep analytical balances and other work areas clean; deal with spills promptly.
- 4.1.4. When using concentrated acids and bases, keep a fully-stocked spill kit (including suitable neutralizers and absorbents) in the work area at all times.
- 4.2. Maintain a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals specified in this method.
- 4.3. Maintain a reference file of material safety data sheets (MSDS), which will be available to all personnel involved in these analyses.

5. Cautions

- 5.1. Considerable heat is generated when dissolving sodium hydroxide (NaOH) in water, or when mixing acid with water. Cool the container in an ice bath when preparing NaOH solutions, or when mixing acid with water.
- 5.2. Excessive foaming during distillation can cause foam to pass into the condenser, which can contaminate the receiving flask and condenser with alkaline salts. Treat heavily-foaming samples with an anti-foaming agent.

6. Interferences

- 6.1. Keep samples at or below 4°C after collection and during shipping (SOP M1), and prior to analysis, to minimize microbial activity. Analyze all samples within 7 days of collection. Frozen (archived) samples can be maintained indefinitely.
- 6.2. Use only ammonia-free water, prepared by passing reagent water through an ion-exchange column packed with strongly acidic cation- and strongly basic anion-exchange resins. Regenerate this column periodically according to the manufacturer's instructions.
- 6.3. Ammonia gas in the lab can deposit into the sample. Minimize sample exposure to air.
- 6.4. Nitrate in large quantities (>10 mg/L) can oxidize ammonia in the sample and introduce negative error. Although this interference cannot be prevented, its effect can be predicted and accounted for on the basis of preliminary nitrate determination of the sample. Whenever high nitrate levels are a potential issue, conduct nitrate analyses of the sample before attempting an ammonia analysis.
- 6.5. Volatile nitrogen-containing compounds such as hydrazine and some amines can cause overestimates of ammonia content. The levels of these in manure should be negligible.
- 6.6. Any residual Cl⁻ in the sample must be removed using sodium thiosulfate. This is most often associated with aqueous samples, or samples containing free water, and generally does not affect manure/biosolid samples.

7. Personnel Qualifications

- 7.1. Personnel should be trained in conducting these analyses before initiating the procedure.
- 7.2. Each analyst must read and understand this SOP, SOP M3, and USEPA Methods 351.3

and 1687 before initiating this procedure, and must show the ability to generate acceptable data accuracy and precision (Section 11.3).

8. Equipment and Supplies

- 8.1. Copies of PAAQL SOP M3 and USEPA Methods 351.3 and 1687
- 8.2. Foss Kjeltex Model 2300 automated distillation and titration analyzer (FOSS Analytical, Hilleroed, Denmark), or equivalent
- 8.3. Flasks: 100-ml Kjeldahl and 50-ml Erlenmeyer flasks
- 8.4. Analytical balance (Capacity = 410 g; Accuracy = 0.001 g)
 - 8.4.1. Reference weights for calibration: 2-mg, 1000-mg, and 50-g class "S" weights
- 8.5. Reagents and buffers
 - 8.5.1. Sulfuric acid: Concentrated, 0.020N & 0.25N H₂SO₄, certified (Midland Scientific, Omaha, NE)
 - 8.5.2. Sodium hydroxide: 40% NaOH solution, certified (Midland Scientific, Omaha, NE)
 - 8.5.3. Boric acid indicator:
 - 8.5.3.1. Add 0.10 g bromocresol green, 0.10 g methyl red, 100 g boric acid, and 20 mL of 0.10N NaOH to a 2-L flask that is half-full of NH₃-free water. Dissolve all reagents
 - 8.5.3.2. Add NH₃-free water to the 2-L mark on the flask.
 - 8.5.3.3. Quantitatively transfer flask contents to the receiving solution reservoir below the Foss Kjeltex Model 2300.
 - 8.5.3.4. Add 8 more liters of NH₃-free water to the reservoir, for a total of 10 L of solution.
- 8.6. Standards
 - 8.6.1. Ammonia standards
 - 8.6.1.1. Ammonia stock solution (1000 mg/L NH₃-N):
 - 8.6.1.1.1. Dry anhydrous ammonium chloride (NH₄Cl) at 105°C immediately before use.
 - 8.6.1.1.2. Dissolve 0.382 g of this dried NH₄Cl in NH₃-free water and dilute to 100 mL with NH₃-free water in a volumetric flask.
 - 8.6.1.2. Ammonia working standard (10 mg/L NH₃-N): Dilute 10 mL of ammonia stock solution to 1 L in a volumetric flask with NH₃-free water.
 - 8.6.2. Calibration standards
 - 8.6.2.1. Prepare calibration standards by spiking 5-g aliquots of blank sand or diatomaceous earth (Section 8.7) directly with ammonia standard solution (Section 8.6.1).
 - 8.6.2.2. Prepare a minimum of five calibration standards, at five different concentrations to produce a calibration curve with at least five points.
 - 8.6.2.3. At least one of the calibration standards must correspond to a sample concentration at or below that necessary to meet the data quality objectives (DQOs) of the project. The QAPP for the particular project will state what concentration meets the DQOs.

- 8.6.2.4. The remaining standards should correspond to the concentration range found in actual samples, without exceeding the titration-based system's working range.
- 8.6.3. Calibration Verification (CV) standard
 - 8.6.3.1. Prepare the CV standard so its NH_3 concentration approximates the midpoint of the calibration curve.
 - 8.6.3.2. Prepare solid CV standards for use with solid samples, and slurry CV samples for use with slurry samples.
 - 8.6.3.3. The source used to prepare the CV standard should be different from that used to prepare the calibration standards. Use either a different bottle of the same compound, or a different NH_3 -containing compound altogether.
 - 8.6.3.4. If a different ammonium compound is used for the CV stock, adjust the amount weighed according to the ratio of NH_3 -N atomic weight to molecular weight. Ammonium chloride has one NH_4 group per molecule and a formula weight of 53.49; it is, therefore 31.78% NH_3 by weight (17/53.49). If, for example, dibasic ammonium phosphate (2 NH_4 per molecule, formula weight 132.06, 25.75% NH_3) is used as the CV standard, and X mg of ammonium chloride was needed to yield the NH_3 concentration at the midpoint of the calibration curve, then $(31.78/25.75) X$, or 1.23 X g of dibasic ammonium phosphate would be needed to yield the same NH_3 concentration.
- 8.6.4. Quality Control Sample (QCS): A prepared quality control sample from a standards vendor. Environmental Resource Associates (Arvada, CO) Catalog #545, or equivalent.
- 8.7. Blank sand or diatomaceous earth
 - 8.7.1. Bake 500 g diatomaceous earth or clean sand at 400 °C for 8 h. Cool and store in a glass container with a sealing lid.

9. Procedures

9.1. Sample Preparation

- 9.1.1. Obtain percent total solids of each sample, according to SOP M3.
- 9.1.2. Label Kjeldahl distillation flasks as necessary to accommodate samples, in duplicate, as well as all necessary duplicates, Precision and Recovery Samples, Calibration Verification Standards, Quality Control Standards, Matrix Spikes, and/or Blanks (See Sections 11.4 - 11.9 for the frequency that these must be included).
- 9.1.3. Add sample.
 - 9.1.3.1. Solid samples: Bring to room temperature. Thoroughly homogenize the sample. Weigh 1-2 g of sample into the Kjeldahl flask.
 - 9.1.3.2. Slurry samples: Bring to room temperature. Mix the sample thoroughly. Transfer (by pipette or spatula) 1-2 mL (or 1-2 g) of sample to the distillation flask, and record the weight of the added sample.

9.2. Distillation

- 9.2.1. Place samples on the Model 2300 and select "Kjeldahl 5". This selection ensures that the instrument will dispense a sufficient amount of NaOH to the sample.
 - 9.2.1.1. Use 0.020 N H_2SO_4 for low-N samples, and 0.25 N for all others. Most samples associated with livestock manure will require 0.25 N H_2SO_4 .

9.3. Calculations

- 9.3.1. Calculate NH₃-N (in units of mg/g) in the sample as follows, correcting the amount of titrant (0.25N H₂SO₄) added to each sample by subtracting that needed to titrate a blank to endpoint:

$$\text{NH}_3\text{-N} = \frac{\text{Titrant}_{\text{sample}} (\text{mL}) - \text{Titrant}_{\text{blank}} (\text{mL})}{\text{Sample wt (g)}} \times 3,500^*$$

*If 0.25N H₂SO₄ is used in the distillation/titration step. If 0.02 N acid is used, replace 3,500 with 280.

10. Data and Records Management

- 10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
- 10.2. Manage all data according to SOP B5.
- 10.3. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes. Overstrike all errors in writing with a single line.
- 10.4. Initial and date all corrections.

11. Quality Control and Quality Assurance

11.1. Reagent Purity

- 11.1.1. All water used in this method must be NH₃-free. See Step 6.1 for the methodology used to remove NH₃ from water for use in these procedures.
- 11.1.2. All other reagents will be ACS Reagent Grade or better.

11.2. Instrument calibration and standardization

- 11.2.1. Calibrate the analytical balance annually (at a minimum).
- 11.2.1.1. Calibrate at 2 mg and 1000 mg using class "S" weights.
- 11.2.1.2. Instrument calibration must be within ±10% at 2 mg (i.e. ±0.2 mg) and ±0.5% at 1000 mg (i.e. ±5 mg). If these limits are not achieved, the balance must be re-calibrated.
- 11.2.1.3. Place a 50-g weight and the 2-mg weight on the balance together. Verify that the balance reads 50.002 g (±0.0002 g).

11.3. Initial demonstration of analyst & laboratory proficiency

- 11.3.1. The following steps are to be followed by every new analyst performing this method, in order to provide an assurance of the quality of his/her technique. No analyst may perform analyses of actual samples until he/she has passed all of the following:

- 11.3.1.1. Determine Method Detection Limit (MDL)
 - 11.3.1.1.1. Prepare a group of at least seven (7) MDL determination samples by mixing 5-g aliquots of blank sand (or diatomaceous earth) (Section 8.7) with 10 mL NH₃-free water, and spiking each with ammonia standard (Section 8.6.1) such that the organic NH₃ concentration of each MDL sample is one to five times the estimated detection limit (Section 1.3.1).
 - 11.3.1.1.2. Using the seven replicates; process each through every step of the method. Calculate the NH₃ concentrations (Section 9.3) for each aliquot, and compare with the expected concentrations.
 - 11.3.1.1.3. If, on average, the calculated concentrations are within 20% of the expected concentrations, analyst and system performance are acceptable.
- 11.3.1.2. Initial Precision and Recovery (IPR)
 - 11.3.1.2.1. Prepare a group of at least four (4) IPR samples by mixing 5-g aliquots of blank sand (or diatomaceous earth) with 10 mL NH₃-free water, and spiking the slurries with ammonia standard such that the NH₃ concentration of each PR sample is on the order of 1%. Process each sample through every step of the method. Perform the % NH₃ calculations in Section 9.3, and determine the percent recovery by comparing these to the known input amount.
 - 11.3.1.2.2. Using the results of the four analyses, compute the average percent recovery (X) and the standard deviation (s) of the percent recovery for ammonia.
 - 11.3.1.2.3. The required performance specifications for PR samples are a recovery of $110\% \geq X \geq 90\%$, and precision (s) $\leq 5\%$.
 - 11.3.1.2.4. If s and X meet the acceptance criteria, analyst and system performance are acceptable.
 - 11.3.1.2.5. If s exceeds the precision limit (5%), and/or X falls outside the range for recovery, system performance is unacceptable. In this event, correct the problem, and repeat the test.
- 11.3.1.3. Blank
 - 11.3.1.3.1. Analyst will include a minimum of one blank (Section 11.9) to verify that he/she does not detect NH₃ in the blank at a concentration greater than the stated MDL (Section 1.3.1).
- 11.3.1.4. Linearity of calibration curve
 - 11.3.1.4.1. Analyst will prepare a five-point calibration curve (Section 8.6.2) to verify his/her ability to generate linear results over the concentration range of interest. If the linear correlation coefficient (r^2) of the calibration curve is ≥ 0.96 , analyst and system performance are acceptable.
- 11.3.1.5. CV Standard
 - 11.3.1.5.1. Analyst will include a minimum of one CV Standard (Section 8.6.3) to verify that he/she can meet acceptance criteria (Section 11.6.1.1).
- 11.4. Ongoing Precision and Recovery (OPR)
 - 11.4.1. MDL
 - 11.4.1.1. Re-determine/verify method detection limits annually.
 - 11.4.1.2. Re-determine/verify method detection limits whenever a modification is made to the method or analytical system.

11.4.2. Precision and Recovery (PR) Samples

11.4.2.1. Each batch (up to 20 samples) (Section 3.11) must contain a minimum of one PR sample. Run solid samples with solid PR samples, and slurry samples with slurry PR samples.

11.4.2.1.1. Prepare a solid PR sample by spiking 5 g of blank sand (or diatomaceous earth) with ammonia working standard such that the NH_3 concentration of the PR sample is approximately five times the MDL (Section 1.3.1).

11.4.2.1.2. Prepare a slurry PR sample by mixing 5 g of blank sand (or diatomaceous earth) with 10 mL NH_3 -free water, and spiking the slurry with ammonia working standard such that the NH_3 concentration of the PR sample is approximately five times the MDL (Section 1.3.1).

11.4.2.2. Carry the PR sample through the entire analytical process, and calculate percent recovery.

11.4.2.2.1. If recovery falls within the range of 90% to 110%, the system performance is acceptable.

11.4.2.2.2. If recovery falls outside the range of 90% to 110%, the system performance is unacceptable, and the problem must be identified and resolved before performing any further analyses.

11.4.2.3. All samples must be associated with an acceptable PR standard before their results may be reported.

11.5. Calibration curves

11.5.1. Generate a new calibration curve (containing a minimum of five points as described in Section 8.6.2) whenever a new standard is obtained.

11.6. Calibration Verification Standards

11.6.1. Run a Calibration Verification Standard, either solid or slurry as appropriate based on the sample matrix, once per sample batch (every 20 analyses – Section 3.11).

11.6.1.1. Acceptance criteria for the CV Standard is $100\% \pm 10\%$.

11.6.1.2. If the CV does not meet the acceptance criteria, the problem must be identified and corrected, including possible recalibration of the system if the problem cannot be corrected by other means.

11.6.1.3. All samples must be associated with an acceptable CV Standard before their results may be reported.

11.7. Quality Control (QC) Standards

11.7.1. In accordance with Method 1687, analyze a QC standard with each day's distillations, or every 12 h, whichever is more frequent.

11.8. Matrix Spikes (MS)

11.8.1. To assess the performance of the method on a given sample matrix, spike, in duplicate, a minimum of 5% (one sample in 20) of the samples from a given sampling site.

11.8.2. The concentration of NH_3 -N spiked into each MS sample shall be 1-5 times the background concentration for the corresponding batch of samples.

11.8.2.1. The spiking level may be established *a priori* based on existing information (either analyst's previous experience or existing literature values) for NH_3 content of manures from the particular species.

11.8.3. The concentration added should be expressed as mg/kg, and should be calculated for a 1-g aliquot by multiplying the N concentration (mg/L) added (as ammonium chloride) by the conversion factor 100 (mg/L x 0.1L/0.001kg = 100).

11.8.4. Do not use blanks for MS analyses.

11.8.5. Assessing spike recovery

11.8.5.1. Calculate the percent recoveries of NH₃-N and relative percent difference (RPD) of the duplicate matrix spike samples (corrected for the background concentration measured in the corresponding unspiked sample which was used as the basis for the MS samples).

11.8.5.1.1. Calculate percent recovery as follows:

$$\text{Percent Recovery} = \frac{(C_s - C_b)}{S} \times 100$$

Where:

C_s = Sample NH₃-N concentration determined after spiking
 C_b = Sample NH₃-N concentration determined before spiking
 S = Known concentration of the spike

11.8.5.1.2. Calculate RPD as follows:

$$\text{RPD} = \frac{(|D_1 - D_2|)}{D_1 + D_2} \times 100$$

Where:

D_1 = NH₃-N concentration of MS sample #1
 D_2 = NH₃-N concentration of MS sample #2

11.8.5.2. Percent recoveries for the matrix spikes should be within three standard deviations of the mean value, with RPD ≤ 20%.

11.8.5.3. If either the percent recovery or the RPD of NH₃-N in the MS samples falls outside the designated range, while the percent recovery in the PR sample is within the acceptable range, the problem encountered with the MS sample is judged to be matrix-related instead of method-related.

11.9. Blanks

11.9.1. Run a minimum of one blank with each batch of 20 samples (Section 3.11).

11.9.2. Suitable blanks are 10 mL of NH₃-free water, or 5 g of blank sand.

11.9.3. If NH₃-N is detected in the blank at a concentration greater than the MDL stated in Section 1.3.1, halt analysis of samples until the source of contamination is eliminated and a new blank shows no contamination.

11.9.4. All samples must be associated with an uncontaminated laboratory blank before their results may be reported.

11.10. QC Charts

11.10.1. Track all IPR, OPR, Calibration Verification, QC Standard, MS and Blank results to provide graphical representations of continued laboratory performance.

11.11. Method Modifications & Re-validation

11.11.1. Each time any modification is made to this method, the analyst will verify that the modified method meets or exceeds the Method Detection Limit and Initial Precision and Recovery achieved by the same analyst using the original (unmodified) method.

11.11.2. The laboratory will maintain records of any modifications made to the method. At minimum, these records must include the following:

- 11.11.2.1. Names, titles, and contact information for analyst who initiated the modifications and for the QA Officer who witnessed and approved them
- 11.11.2.2. Narrative stating reason(s) for the modification
- 11.11.2.3. Results from all QC tests comparing the modified method's performance to that of the original method:
 - Initial Precision and Recovery
 - Ongoing Precision and Recovery
 - Results with Calibration Verification, QC Standard, MS samples and Blanks

11.12. Replication

11.12.1. Run duplicates of 10% of the samples in each batch, with a minimum of one duplicate per batch. Initial masses (i.e. wet weights) of the duplicates will not differ by more than 10%. Replicates must agree to within 10% of their average NH_3 content; if not, the batch of samples must be re-run.

12. References

- 12.1. Bremner, J.M. and D.R. Keeney. 1965. Steam distillation methods for determination of ammonium, nitrate and nitrite. *Analytica Chimica Acta*. 32:485-495.
- 12.2. Peters, J., A. Wolf, and N. Wolf. (2003). "Ammonium Nitrogen". Recommended Methods of Manure Analysis (J. Peters, ed.). University of Wisconsin-Madison Cooperative Extension Publishing, pp. 25-29. Online at <http://cecommerce.uwex.edu/pdfs/A3769.PDF>; accessed 1/18/2006.
- 12.3. Hoskins, B. 2003. "Laboratory Procedures". Recommended Methods of Manure Analysis (J. Peters, ed.). University of Wisconsin-Madison Cooperative Extension Publishing, pp. 12-13. Online at <http://learningstore.uwex.edu/Recommended-Methods-of-Manure-Analysis-P106C0.aspx>. Accessed 9/6/2007.
- 12.4. USEPA Method 351.3. 1978. Nitrogen, Kjeldahl (Colorimetric, Titrimetric, Potentiometric). Online at http://www.umass.edu/tei/mwwp/acrobat/epa351_3Norg.pdf. Accessed 8/6/2007.
- 12.5. USEPA Method 1687. 2001. Total Kjeldahl Nitrogen in Water and Biosolids by Automated Colorimetry with Preliminary Distillation/Digestion (Draft). EPA-821/R-01-004 (CD). USEPA Office of Water, Office of Science and Technology. Washington, DC.

- 12.6. USEPA Method 1690. 2001. Ammonia-N in Water and Biosolids by Automated Colorimetry with Preliminary Distillation (Draft). EPA-821/R-01-009 (CD). USEPA Office of Water, Office of Science and Technology. Washington, DC.
- 12.7. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.

**MEASUREMENT OF BARN INLET AND EXHAUST PARTICULATE
MATTER (TSP, PM10, PM2.5) CONCENTRATIONS USING THE
THERMOFISHER SCIENTIFIC - SOP P1**

**MEASUREMENT OF BARN INLET AND EXHAUST PARTICULATE MATTER
(TSP, PM₁₀, PM_{2.5}) CONCENTRATIONS USING THE THERMO ELECTRON
(RUPPRECHT AND PATASHNICK) TEOM SERIES 1400A PM MONITOR**

Standard Operating Procedure (SOP) P1

**Prepared by
Bill W. Bogan and Teng Teeh Lim**

**Reviewed by
Lingying Zhao (Ohio State Univ.) and Albert J. Heber**

Effective Date: November 6, 2006

**PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)
Agricultural and Biological Engineering, Purdue University
West Lafayette, IN 47907**

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions of Terms and Acronyms	3
4. Health and Safety	4
5. Cautions.....	4
6. Interferences	5
7. Personnel Qualifications	5
8. Equipment and Supplies	5
9. Procedures.....	6
10. Data and Records Management.....	11
11. Quality Control and Quality Assurance Section	11
12. References	13
13. Contact Information.....	13
Appendix A. Design and Specifications of TEOM Cabinets.....	14

1. Scope and Applicability

- 1.1. The Rupprecht and Pataschnick (R&P) TEOM 1400a monitor will be used for continuous monitoring of the total suspended particle (TSP), PM₁₀, (particulate matter of 10 microns and smaller), and PM_{2.5} concentrations, of ambient air and in the exhaust air of confined animal houses or barns. The TEOM instrument is a continuous PM-monitoring device designated by USEPA as an equivalent method (EPA Designation No. EQPM-1090-079) for PM₁₀.
- 1.2. Measurement range of the TEOM 1400a is 0 to 5,000,000 µg/m³, with a minimum detection limit of 0.06 µg/m³ (1-h average).
- 1.3. Use of the TEOM 1400a is appropriate for air temperatures in the range of -40 to 60°C, although the sensor and control units must be maintained between 2 and 40°C. If field temperatures outside this range are expected, this is accomplished by keeping the sensor and control units inside the on-farm instrument shelter (OFIS).
- 1.4. This procedure applies only to the Thermo Electron TEOM 1400a PM Monitor.

2. Summary of Method

The TEOM 1400a PM monitor works by extracting air into an inlet head that inertially separates PM₁₀ particles, and collects a portion of the PM₁₀ particles onto a Teflon-coated glass fiber filter. A sensitive mass transducer determines the mass of PM accumulated on the filter by measuring the change in vibratory frequency in a hollow, tapered element that holds the filter cartridge. Changes in the resonant frequency are sampled electronically in quasi-real time, providing both continuous and time-averaged measures of mass accumulation that are directly proportional to instantaneous and time-averaged mass concentrations in air, respectively. The instrument samples the vibratory frequency of the element every two seconds and the instrument microcomputer calculates the new system mass (element + filter + collected PM). The device operates at an industry-standard, volume-controlled flow rate of 16.7 L/min. The sample stream is preheated to 50°C before entering the mass transducer so that the filter always collects under conditions of very low (and therefore relatively constant) humidity.

3. Definitions of Terms and Acronyms

- | | | |
|------|--------------------|---|
| 3.1. | AK | Standard protocol for communication among instrumentation. It is used by the RP _{COMM} software for internal data logging. |
| 3.2. | A/O | Analog output |
| 3.3. | atm | atmosphere, 1013 mBar |
| 3.4. | Mass Concentration | Mass of substance per unit volume of gas |
| 3.5. | OFIS | On-farm instrument shelter |
| 3.6. | PAAQL | Purdue Agricultural Air Quality Laboratory |
| 3.7. | PCB | Printed circuit board |
| 3.8. | PM | Particulate matter |
| 3.9. | PM ₁₀ | PM of 10 microns diameter and under |

3.10. PM _{2.5}	PM of 2.5 microns diameter and under
3.11. R&P	Rupprecht and Patashnick
3.12. RH	Relative humidity
3.13. TEOM	Tapered Element Oscillating Microbalance
3.14. TSP	Total suspended particle or total suspended particulate (matter)

4. Health and Safety

- 4.1. Be careful when working with electrical power connections. Repair of instruments should only be carried out by trained personnel, and in accordance with procedures detailed in the instrument manual.

5. Cautions

- 5.1. When sampling the exhaust air stream, locate the TEOM PM₁₀ inlet near the inlet of the fan (or upstream of the fan), however, far enough away to avoid concerns about anisokinetic sampling. As a safe precaution, the air velocity around the sampling head should be 2 m/s (400 ft/min) or less. This corresponds to the designed air velocity of a tunnel-ventilated barn.
- 5.2. Protect the instrument from exposure to harsh environments with the outdoor enclosure unit. However, the outdoor enclosure unit may be too large for most exhaust air sampling locations in the barn. If the sampling location is 20 m or less from the on-farm instrument shelter, place the sampling pump and control unit in the OFIS (On-Farm Instrument Shelter) and protect the sensor unit with a custom-made enclosure (See Appendix A).
- 5.3. Prevent damage by rodents, flies and animals. All the openings of the enclosure should be sealed. Mothballs can be used inside the enclosure; however, adverse effects of the performance of a TEOM equipped with a filter dynamics measurement system (FDMS) in the presence of mothballs has been reported. The instrument should be isolated from moisture and manure, and mechanical vibration. Locate the sensor unit where external objects are not likely to disturb the instrument enclosure or the air sampling tube.
- 5.4. Protect instrument from adverse weather, especially precipitation. When the instrument control unit is installed on top of the enclosure unit, be certain the sampling tube leading out of the building or enclosure has a watertight fit.
- 5.5. Ensure that adequate power is available at the site. Lack of a stable power source can result in the loss of data because of power interruptions. The system resets itself every time when power is regained, requiring a period of stabilizing before providing valid data. An uninterruptible power supply with battery backup should be used to prevent damage and data loss in case of power failure. Furthermore, a lightning arrestor will protect the entire on-farm instrument shelter and secondary surge suppressors will protect the PC and the instruments from electrical surges.
- 5.6. The TEOM is a complex instrument and requires some special training (Section 7) in its use and operation to obtain consistently good data. Although it is capable of automatic operation, the menu structure is complex and is easily misused.

6. Interferences

- 6.1. Relative humidity fluctuations of the sample stream may cause measurement accuracy to decrease due to the inlet system's inability to adjust the temperature to that specified by the software.
- 6.2. Mechanical vibration which is transferred to the instrument can interfere with the vibrational frequency of the TEOM's tapered element, and can therefore lead to inaccurate measurements.
- 6.3. The sensor inlet should be directly below the flow splitter. Improperly locating the sensor inlet relative to the flow splitter can cause particulate to settle out of the air stream and collect on the tubing walls prior to reaching the filter, thus yielding inaccurate results.
- 6.4. Dust buildup on the external screen of the PM₁₀ Inlet can trap particulate matter, preventing it from reaching the sensor.

7. Personnel Qualifications

- 7.1. Personnel should be trained in the use of the instrument before initiating the procedure. Training requires about four hours.
- 7.2. Each analyst must read and understand the entire instrument manual and this SOP before operating the instrument.
- 7.3. Personnel should also be trained to interpret the analyzer output signal (including the interference by relative humidity), set up the internal data logger, and interpret the selected logged parameters.

8. Equipment and Supplies

- 8.1. TEOM Series 1400a ambient particulate monitor (Thermo Electron Corporation, East Greenbush, NY)
- 8.2. TEOM sensor unit
- 8.3. TEOM PM_{2.5} inlet
- 8.4. TEOM PM₁₀ inlet
- 8.5. TEOM TSP inlet
- 8.6. TEOM flow splitter assembly
- 8.7. Filter exchange tool
- 8.8. Electric and air cable assembly for connecting the control unit to the sensor unit
- 8.9. Consumables to be kept on hand at each monitoring site include the following:
 - 8.9.1. One box of 20 TX40 TEOM filters (R&P #57-007225-0020)
 - 8.9.2. Four large bypass in-line filters (R&P #57-002758)
 - 8.9.3. One piston pump rebuild kit (R&P #59-008630)
- 8.10. The following spare parts (one each) should be kept on hand at PAAQL for a network of 10 TEOMs:
 - 8.10.1. Frequency counter printed circuit board (PCB) (R&P #10-000394)
 - 8.10.2. Main computer PCB (R&P #10-001594-0006)

- 8.10.3. Analog I/O PCB (R&P #57-001595-0016)
- 8.10.4. Dual flow controller (R&P #55-007675)
- 8.11. Mass calibration verification kit (R&P #59-008298-0005)
- 8.12. Calibrated 3 ½ digit multimeter & 30 cm (12") jumper wire (for analog board calibration)

9. Procedures

- 9.1. Instrument setup
 - 9.1.1. Check all components carefully to assure they are free of visible defects or damage.
 - 9.1.2. Set up the control and sensor units (Section 2.3 of manual). Ensure that all air tubes are pushed completely into the fitting so that they cannot be pulled out.
 - 9.1.3. Connect analog output (0-5 or 0-10 VDC) of the analyzer to the data acquisition system (SOP B2). The three analog output channels of the instrument are accessible from the identical 15-pin connectors on the front and back panels of the control unit. Details regarding the pin assignments and voltage (VDC) outputs are given in the operating manual (Section 5.1). To bring up the Set Analog Output Screen, press <A/O>, or select "Set Analog Output" from the Menu Screen, or press 04<Enter>.
 - 9.1.4. Route the electric cable and air tubes to the sensor unit so that the cable is protected and the sample and bypass tubing are not kinked. Extensions of the air tube (1/4" or 3/8" diameter) and cables (9-pin for temperature probe or 25-pin for sensor unit) can be used, if needed. The extension cables are straight-through with male-female connectors.
 - 9.1.5. Installing the Sampling System:
 - 9.1.5.1. Set up sampling hardware (Section 2.4 of manual). Ensure that the inner tube of flow splitter is 15.5 cm (6 in) from the open end of the outer tube.
 - 9.1.5.2. If used for ambient monitoring, install the TEOM either with the environmental controlled outdoor unit or on top of an instrument shelter (SOP U2).
 - 9.1.5.3. Use a custom-made small enclosure (Appendix A) for the sensor unit and keep the control unit and sampling pump inside an instrument shelter (Fig. 1).
- 9.2. Installing/Replacing the filter cartridge (Refer to Section 3.2 of Operating Manual for detailed procedures)
 - 9.2.1. Press the <Data Stop> key before opening the door of the sensor unit to stop measurement temporarily.
 - 9.2.2. Store the loaded filters for further analysis or for recycling.
- 9.3. Installing and cleaning the PM_{2.5} sampling inlet
 - 9.3.1. For PM_{2.5} measurement, install the PM_{2.5} inlet over the open end of the flow splitter (Fig. 2).
 - 9.3.2. The sampling inlet requires the use of a PM₁₀ inlet. Install the PM₁₀ inlet onto the air entrance part of the PM_{2.5} inlet.



Figure 1. TEOM sensor unit in a custom-made enclosure.



Figure 2. TEOM PM₁₀ and PM_{2.5} inlets (left), and a closeup of the PM_{2.5} inlet (right, photo provided by TEC).

9.3.3. Cleaning the PM_{2.5} sample inlet:

- 9.3.3.1. Push the <Data Stop> button on the keypad. Remove the PM₁₀ and PM_{2.5} inlets, and replace with an in-line filter to prevent contamination of the inlet tube.
- 9.3.3.2. Detach the transfer tube (the rectangular-shape part on the left, left photo of Fig. 2). If it is too tight to be removed by hand, pry it off with a rigid plastic lever.

- 9.3.3.3. Remove the top cap and grit pot by unscrewing.
 - 9.3.3.4. Use a wet, lint-free wipe to remove all visible PM deposits. It is recommended to use mineral-free (de-ionized) water and lint-free laboratory wipes, and use ultrasonic cleaning for stubborn deposits. The PM are most likely found at the bottom of the cone and inside the grit pot.
 - 9.3.3.5. After cleaning, check the o-rings, for shape and integrity, and replace if needed. Apply silicone grease as needed to maintain a thin layer on the surfaces around the o-rings.
 - 9.3.3.6. Carefully reassemble all the parts back, and reinstall. Also check and lubricate the transfer tube if needed to ease assembly and prevent leakage.
 - 9.3.3.7. Press <Data Stop> on the controller. This prevents the recording of skewed data.
 - 9.3.3.8. Instrument will automatically resume recording in approximately 30 min. Scheduled inlet cleaning may be combined with the filter exchange to minimize downtime.
 - 9.3.3.9. Clean the PM₁₀ sample inlet following the procedures listed below.
- 9.4. Installing and cleaning the PM₁₀ sample inlet
- 9.4.1. For PM₁₀ measurement, use only the PM₁₀ inlet over the open end of the flow splitter.
 - 9.4.2. Cleaning the PM₁₀ sampling inlet:
 - 9.4.2.1. Similar to the procedures of cleaning the PM_{2.5} inlet, always push the <Data Stop> button before and after the cleaning, and use an in-line filter to prevent contamination of the inlet tube if necessary.
 - 9.4.2.2. Unscrew the acceleration assembly from the collector assembly. Using pressure air or a can of pressurized Dust Remover (available at most hardware and electronics stores), blow the inlet screen on the acceleration assembly until visibly clean. Then blow the inside of the acceleration assembly through the bottom tube. Also blow off the internal areas of the Collector Assembly.
 - 9.4.2.3. If necessary, use a wet and lint-free wipe to clean the surfaces carefully.
 - 9.4.2.4. Check the o-rings, located on the lower part of the Acceleration Assembly and on the lower part of the Collector Assembly, for the presence of silicon grease. Apply silicone grease as needed to maintain a thin layer on the surfaces around the o-rings.
 - 9.4.2.5. Carefully replace the inlet unit, and press <Data Stop> on the controller. This prevents the recording of skewed data.
 - 9.4.2.6. Instrument will automatically resume recording in approximately 30 min. Scheduled inlet cleaning may be combined with the filter exchange to minimize downtime.
- 9.5. Installing and cleaning the TSP sampling inlet
- 9.5.1. For TSP measurement, install only the TSP inlet over the open end of the flow splitter (Fig. 3).
 - 9.5.2. Cleaning the TSP sample inlet:
 - 9.5.2.1. Similarly, always push the <Data Stop> button before and after the cleaning.
 - 9.5.2.2. Remove the TSP inlet from the flow splitter, and blow off deposited PM inside and outside of the inlet, by using a can of pressurized Dust Remover inside.



Figure 3. TEOM PM₁₀ (left) and TSP inlets (right).

- 9.5.2.3. Check the o-rings, located on the lower part of the inlet for integrity and presence of silicon grease. Apply silicone grease as needed.
- 9.5.2.4. Carefully replace the inlet unit.
- 9.5.2.5. Instrument will automatically resume recording in approximately 30 min.
- 9.6. Installing and cleaning external screen for PM₁₀ inlet
 - 9.6.1. A wire-mesh external screen was designed to encircle the PM₁₀ inlet to keep flies and large dust particles out. The additional external screen is made of 8x8 mesh stainless steel wire cloth. The diameter of the screen is about 25 cm (10 in), just slightly bigger than the PM₁₀ inlet, enclosing the entire inlet area (Fig. 4). This external screen requires cleaning every 3 to 7 d, depending on PM concentration.
 - 9.6.2. Carefully remove the external screen, and, in an area downwind of the inlet unit, brush or if needed, clean with water and allow to dry.
 - 9.6.3. After all visible dust has been removed from the external screen, carefully replace it onto the PM₁₀ inlet, so as not to disturb the sensor unit.
- 9.7. Leak check (Section 7.6 of manual). Perform the leak check with NO sample filter attached to the mass transducer, which will prevent accidental damage from occurring to the sample filter cartridge when exposed to the high pressure drop (vacuum) in the sample line that the leak check creates. Leakage flow rates should indicate less than 0.15 L/min for the sample main flow and less than 0.6 L/min for the auxiliary flow or bypass flow with the end of the sample line closed. If not, systematically check plumbing for connector leaks.



Figure 4. TEOM PM₁₀ inlets with external screens installed.

- 9.8. Flow rate audits (Section 8.6 of manual). Use the flow audit adapter with a capped nut for closing the flow splitter bypass line port to check the sample and total flow rates. The volumetric flow rates should be within $\pm 7\%$ (± 0.20 and ± 1.17 L/min for the sample and total flow rates, respectively) of the set points. Large errors in the flow may indicate other sources of error, such as a malfunctioning flow controller, a system leak, or improper temperature and pressure settings.
- 9.9. Mass transducer calibration verification (Section 8.6 of manual). The calibration constant should differ less than 2.5% from the original calibration constant. Notify PAAQL and Thermo Electron if the value is greater than 2.5% as indicated in the manual.
- 9.10. Setting/Checking time and date (Section 4.10 of manual). Check time and date when data is downloaded.
- 9.11. Internal data storage and retrieval (Section 4.11 of manual)
 - 9.11.1. Internally logged data are stored in a circular buffer. To view data from the control unit, press <Store>, and select “View Storage”, or enter 08<Enter> from any screen, or download the data via the RS-232 serial port. The instrument stores the time, date, and station number in each record in addition to the data fields selected by the user. The storage capacity (Fig. 4-11 of manual) is about 1.7 weeks of 8 data fields per record, using a storage interval of 10 min.
 - 9.11.2. Ensure that the communication software is set for the same communication parameters as the instrument. The default and best protocol to use is “AK protocol.” Connect a compatible computer to an RS-232 port using the provided 9-to-9 pin computer cable. Make sure the latest version of the RPSComm software is installed properly onto the computer. Bring up the software and wait for a while or click on the front pages. A small window of the software will display the keyboard and 4-line text display as shown on the front panel of the control unit. Use the “Download storage” command to download stored data in the data logger.
 - 9.11.3. Toggle the instrument between the View Storage Screen and Set Storage Screen with the <Step Screen> key, or by pressing 09<Enter> to gain direct access to the Set

Storage Screen from any other screen. The first eight lines of the Set Storage Screen contain the titles of the variables currently being stored by the data logger (See the Appendix A for the Program Register Codes). Use specific program register codes (Table 1) to change the parameter for each of the channel. “Interval” variable is the number of seconds between successive data in the circular buffer.

Table 1. Some common program register codes

Code	Variable	Units
008	Mass concentration	$\mu\text{g}/\text{m}^3$
009	Total mass	μg
035	Filter loading	%
039	Current main flow	L/min
040	Current auxiliary flow	L/min
041	Status condition	code
057	30-min average mass concentration	$\mu\text{g}/\text{m}^3$
130	Current ambient temperature	$^{\circ}\text{C}$
131	Current ambient pressure	atm

10. Data and Records Management

- 10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet that is designated for this method. Supplement this electronic record with a bound record book designated for the method, which should also contain bound copies of the electronic record.
- 10.2. Manage data according to SOP B5.
- 10.3. Document all data and information on field data sheets, and site logbooks with permanent ink, or in electronic field notes.

11. Quality Control and Quality Assurance Section

- 11.1. Exchange TEOM filter cartridges before the % loading figure reaches 70% (suggested for most tubing length) or when the increase from the new-filter value reaches 50% (e.g. from 35%-new to 85%-loaded). This is necessary because some new filters will show higher loading rates when the tubing is long and the large-in-line filter is loaded with PM. Minimizing the filter loading is necessary because when the filter is heavily loaded, the interference of water on high-frequency PM readings is high, and the mass of PM collected on the filter is sensitive to sudden changes in the humidity of the sample air.
- 11.2. Exchange or install filter cartridges only when the instrument is in the Initialization Mode or Data Stop Mode, or when the instrument is turned off, and not when the TEOM system is taking data (i.e., when the instrument is in the Run Mode).
- 11.3. Replacing and pre-conditioning filters
 - 11.3.1. At a flow rate of 3 L/min, 100% filter loading generally corresponds to a total mass accumulation of approximately 3 to 5 mg of particulate. Filter lifetime at a main flow rate of 3 L/min is generally 21 d at an average PM_{10} concentration of $50 \mu\text{g}/\text{m}^3$.

- 11.3.2. Store filters inside the sensor unit for convenience, and to keep them dry and warm. There is a storage position for the next filter available on the mass transducer that keeps the new filter closer to the sampling conditions. Two posts are along the right side of the mass transducer when open.
- 11.3.3. Do not handle filters with fingers. Use the filter exchange tool and follow the procedures given in this SOP, and in the operating manual. Never twist the filter or apply sideways force to the tapered element. Minimize the temperature upset to the system when changing filters by keeping the door of the instrument open for the shortest time possible.
- 11.4. If the filter loading percentage is high immediately after placing a new TEOM filter on the mass transducer, or if the lifetime of TEOM filter cartridges becomes noticeably shorter, this usually indicates that the in-line filter in the main flow line needs to be replaced.
- 11.5. If a actual quality control audit is determined to be out of the ranges as specified by the manufacturer, the data since the last audit should be carefully reviewed and invalidated if needed. The factory-recommended schedule of periodic maintenance, as well as the schedule to be adopted for use in livestock buildings, are as follows:

Table 2. Periodic maintenance of the TEOM instrument.

Maintenance item	Factory	This Project	Corrective Action
Clean external screen	-	As necessary	
Clean PM ₁₀ inlet	Upon filter exchange	Weekly	
Exchange in-line filters	6 month or when loaded	When loaded	
Clean air inlet system	6 month	Bimonthly	
Leak test	Annually	Bimonthly	Locate and repair leak
Flow audit	Annually	Bimonthly	Check for leak and recalibrate
Mass flow controller calibration	Annually	Every 6 month	Recalibrate
Analog board calibration	Annually	Every 6 month	
Mass transducer calibration verification	Annually	Bimonthly	Check system or return to PAAQL

Source: Adapted from Heber et al. (2006)

12. References

- 12.1. Heber, A. J., T.-T. Lim, J.-Q. Ni, P. C. Tao, A. M. Schmidt, J. A. Koziel, S. J. Hoff, L. D. Jacobson, Y. Zhang, and G. B. Baughman. 2006. Quality assured measurements of animal building emissions: Particulate matter concentrations. *Journal of the Air & Waste Management Association* (in press).
- 12.2. Heber, A.J., J.-Q. Ni, T.T. Lim, R. Chervil, P.C. Tao, L.D. Jacobson, S.J. Hoff, J.A. Koziel, Y. Zhang, D.B. Beasley, and J.M. Sweeten. 2005. Aerial pollutant emissions from two high-rise layer barns in Indiana. *A&WMA's 98th Annual Conference & Exhibition, Minneapolis, MN, June 21-24. AWMA, p.15.*
- 12.3. Lim, T.-T., A.J. Heber, J.-Q. Ni, J.Z. Gallien, and H. Xin. 2003. Air quality measurements at a laying hen house: particulate matter concentrations and emissions. *Air Pollution from Agricultural Operations, Durham, NC, USA, October 12-15, pp. 249-256.*
- 12.4. Rupprecht & Patashnick Co, Inc. (2001). TEOM Series 1400a Ambient Particulate (PM-10) Monitor (AB Serial Numbers), Revision B.
- 12.5. SOP B6. 2006. Data Processing Software (CAPECAB). Standard Operating Procedure B6. Purdue Ag Air Quality Lab.
- 12.6. SOP B3. 2006. Air Data Pre-Processing Software. Standard Operating Procedure B3. Purdue Ag Air Quality Lab.
- 12.7. SOP B2. 2006. Data Acquisition and Control Software (AirDAC). Standard Operating Procedure B2. Purdue Ag Air Quality Lab.
- 12.8. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 12.9. SOP U2. 2006. Installation of Barn Measurements. Standard Operating Procedure U2. Purdue Ag Air Quality Lab.
- 12.10. USEPA. 1999. Determination of PM₁₀ in Ambient Air Using a Continuous Rupprecht and Pataschnick (R&P) TEOM® Particle Monitor. Method IO-1.3 in *Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air*. Center for Environmental Research Information, Cincinnati, OH.

13. Contact Information

- 13.1. Manufacturer: Thermo Electron Corporation, 26 Tech Valley Drive, East Greenbush, NY 12061.
- 13.2. Manufacturer Technical Support: 518-452-0065. General e-mail: rpsinfo.eid@thermo.com
- 13.3. PAAQL: odor@purdue.edu, heber@purdue.edu, <http://www.AgAirQuality.com>.

Appendix A. Design and Specifications of TEOM Cabinets.

The vertical rod shown in Figs. A1, A3, and A4 is the mounting rod that holds the inlet pipe of the TOEM sensor unit vertical.

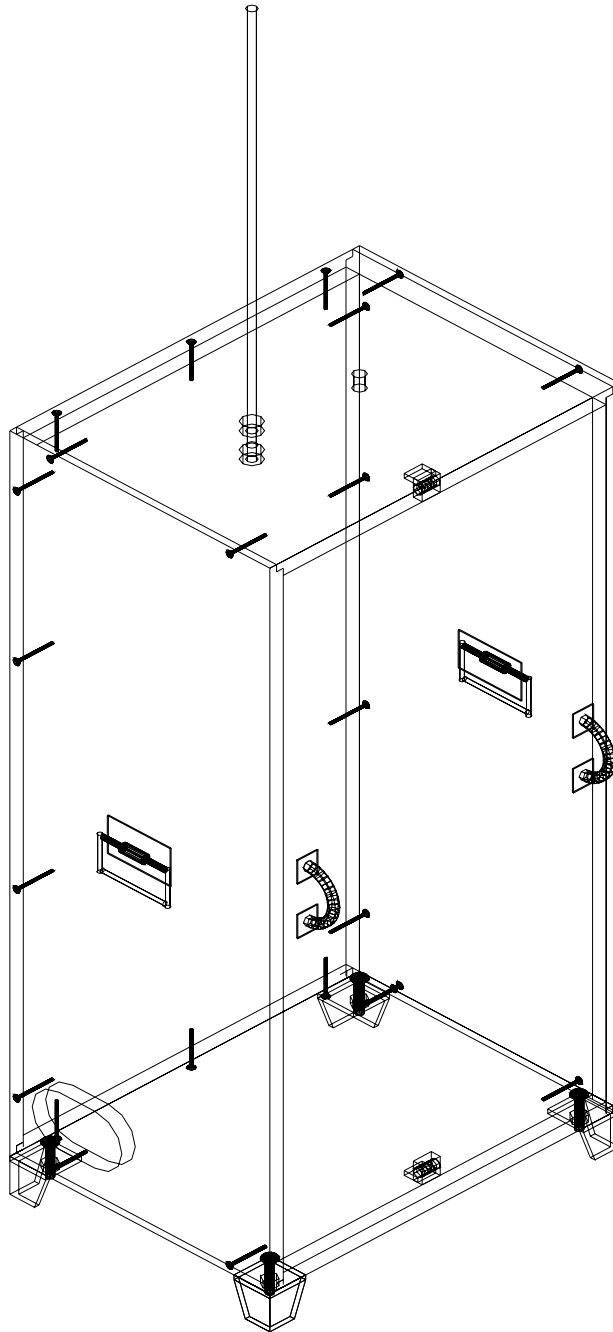


Figure A1. 3-D view.

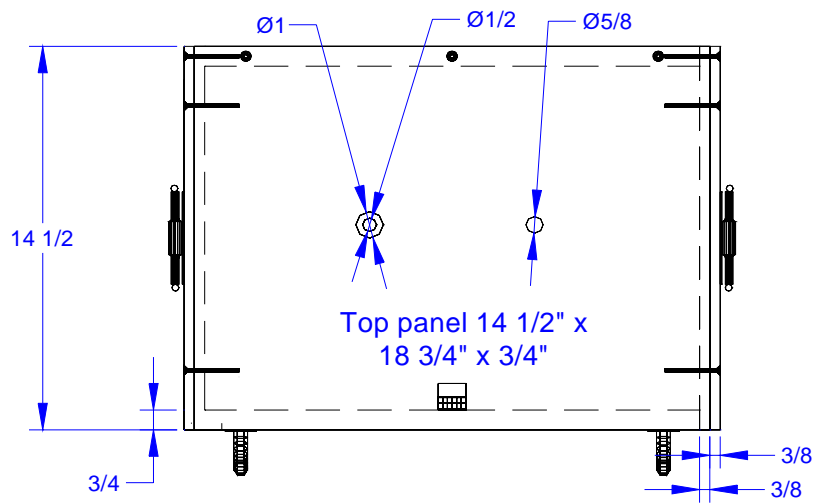


Figure A2. Top view.

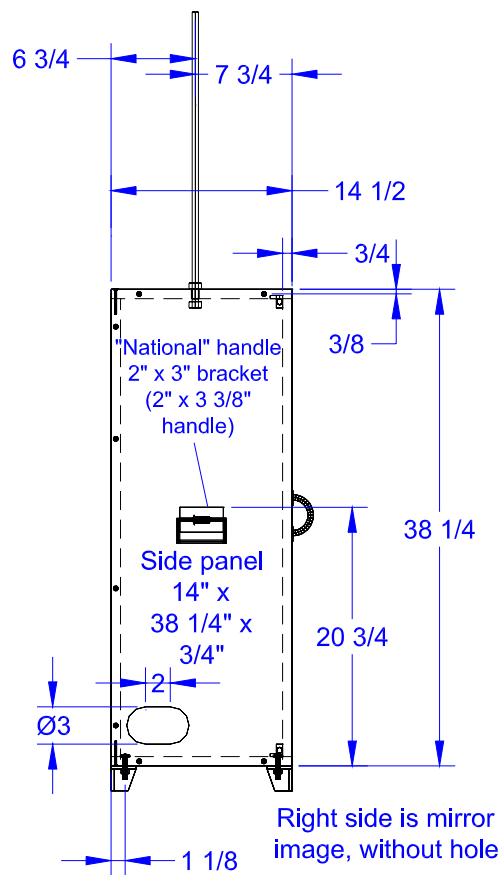


Figure A3. Left side view.

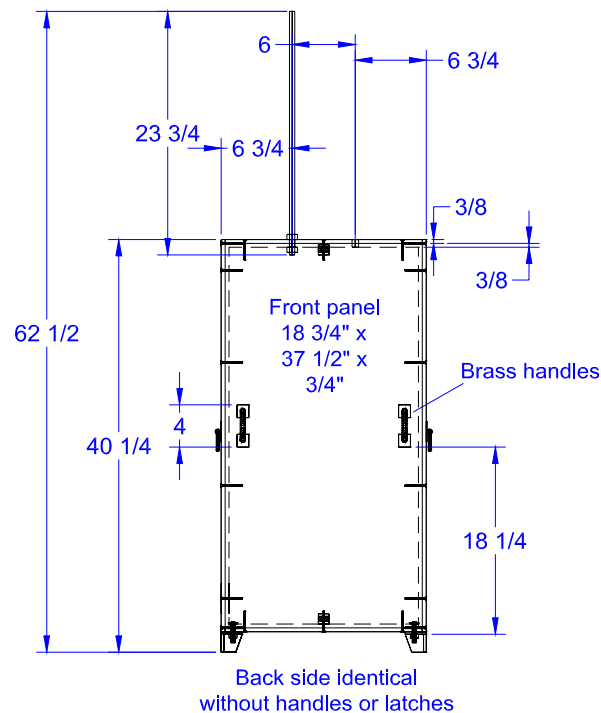


Figure A4. Front view.

Specifications

1. 3/4" plywood (A-C ext.) on all sides.
2. Aluminum feet. 1/4" thick aluminum. 2"x2"x1-15/16" angle reducing to 1-1/2" angle at the bottom welded onto 2"x2" plate which is bolted to plywood.
3. Metal handles on door. National N243-766 170. Sash Lift Brt. Brass finish 4".
4. Metal handles on sides of cabinet. Chest handles. 3-1/2". National N117-002 V175.
5. 40-3/16 inch working height.
6. Top panel is 14-1/2"x19"x3/4".
7. Side panels are 14"x38-1/4"x3/4".
8. Front panel is 18-3/4"x37-1/2"x3/4".
9. Mounting rod is 23.75"x1/2"
10. Mounting rod is 1/2" dia and is threaded at the bottom.
11. Door handles are brass and 4"x1-1/8" and stick out 1".
12. Side handles stick out 3/8".
13. Screws are 2" long #8 drywall screws.
14. One coat of exterior polyurethane.

**MEASUREMENT OF BARN EXHAUST TOTAL SUSPENDED
PARTICULATE CONCENTRATIONS USING AN ISOKINETIC
MULTIPOINT GRAVIMETRIC METHOD**

Standard Operating Procedure (SOP) P2

**MEASUREMENT OF BARN EXHAUST TOTAL SUSPENDED PARTICULATE
CONCENTRATIONS USING AN ISOKINETIC MULTIPOINT
GRAVIMETRIC METHOD**

Standard Operating Procedure (SOP) P2

Prepared by

Juan Carlos Ramirez and Teng-Teeh Lim

Reviewed by

**Bill W. Bogan, Yuanhui Zhang (Univ. of Illinois at Urbana-Champaign),
and Albert J. Heber**

Effective Date: November 6, 2006

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions	4
4. Health and Safety	4
5. Cautions	4
6. Interferences	5
7. Personnel Qualifications	5
8. Equipment and Supplies	6
9. Procedures	6
10. Data and Records Management	12
11. Quality Control and Quality Assurance	12
12. References	13
13. Manufacturer's Contact information	13

1. Scope and Applicability

- 1.1 This procedure presents the methodology and instrumentation to measure total suspended particulate (TSP) concentration in the exhaust air of confined animal houses using an isokinetic multipoint system.
- 1.2 An integrated isokinetic gravimetric sampling system using critical flow rate control was developed by the University of Illinois at Urbana-Champaign (UIUC) and has been used since 1999 (Wang and Zhang, 1999; Jerez et al., 2006), and can be purchased via CCC Technologies.
- 1.3 Sampling is typically conducted at the minimum ventilation fan that is continuously operating at full speed.
- 1.4 The sampling duration is typically 24 h or a multiple of 24 h, depending on the PM concentration. Higher PM concentrations load the filter more rapidly, requiring shorter sampling times.
- 1.5 The sampling flow rate is 20 L/min \pm 10% for each of the three sampling nozzles, which are positioned vertically along the height of the fan and pointing into the direction of the exhaust air stream.

2. Summary of Method

The system which is used for TSP sampling consists of a vacuum pump, three critical venturi and three isokinetic sampling heads, polyethylene (PE) sampling tubes and fittings, and filters. The TSP system samples at the inlets of the exhaust fans using the critical venturi method (Jerez et al., 2006). Each conical isokinetic sampling head is attached to a 37-mm open-faced filter cassette, which is fitted with conditioned and weighed filters. The sampling nozzles should be located at three representative heights, which are carefully selected by using a portable anemometer to match the airstream velocity for isokinetic sampling. The isokinetic conditions are achieved by adjusting the positions of the sampling nozzles near the fan until the air speed at each nozzle inlet is approximately 2 ± 0.2 m/s. Different sizes of the sampling nozzle may be used to match the sampling velocity requirement. TSP sampling can also be conducted without an isokinetic sampling head; this is done in very low air speeds (lower than 0.3 m/s) or calm air sampling conditions. Following EPA Method 17 procedures for recovering PM from stack sampling probes, the inside surfaces of the nozzles are rinsed with acetone, and brushed with a nylon bristle brush. TSP concentrations are determined by combining the mass of TSP collected in the acetone rinse and the desiccated filters, and dividing the combined mass by the total sample air volume. This procedure includes descriptions of how to condition, install, and retrieve the filters for their initial and final weighing and storage.

3. Definitions

3.1	Isokinetic	Air velocity at inlet tip exactly matches surrounding velocity, such that streamlines do not converge into the inlet or diverge around the inlet.
3.2	OFIS	On-farm instrument shelter
3.3	PAAQL	Purdue Agricultural Air Quality Laboratory
3.4	PE	Polyethylene
3.5	PM	Particulate matter
3.6	QAPP	Quality Assurance Project Plan
3.7	SOP	Standard operating procedure
3.8	TSP	Total suspended particulate.
3.9	UIUC	University of Illinois at Urbana-Champaign

4. Health and Safety

- 4.1. The system has a pump operating at 120 VAC. Be careful when working with the electrical power connections, especially if located in wet or moist conditions.
- 4.2. Be careful when working near the ventilation fan. The high-speed fan blades can be a hazard, especially if they are not protected by fan shutters.

5. Cautions

- 5.1. Protect the sampling system from exposure to harsh environments by storing the sampling pump in the OFIS or in an enclosure unit near the sampling location. Prevent damage by rodents, flies and animals. The sampling system should be isolated from moisture and manure. Locate the system where external objects are not likely to disturb the pump or the sampling system.
- 5.2. When the vacuum pump is kept in the OFIS, make sure the long tube between nozzles and pump does not create a pressure drop that affects the critical pressure of the venturi. Provide adequate heating to the vacuum tube to prevent condensation.
- 5.3. Ensure adequate and continuous power is available for the sampling pump. Lack of a stable power source can result in the loss of quality data because of power interruptions. The system is not equipped with a sampling flow rate monitoring device; thus, any power interruption will not be detected or recorded. For example, if the power supply is part of the automated lighting system, which is shut off in the evening, the sampling is then interrupted, although the system appears to be running continuously. Use a vibration sensor (See SOP A7), mounted on the pump, to record pump operations.
- 5.4. Verify and record any change in the internal disposition that affects the flow pattern in the sampling area. If something changes (for example, the height of the manure after a cleanup, fan maintenance or cleaning), adjust the sampling head locations to meet the isokinetic conditions if the sampling is used with sampling nozzles.
- 5.5. Verify that the glass fiber filter is properly sitting on the bottom piece of the filter cassette, such that all of the sampling air must go through the filter. Make sure that the

filter does not move freely after the middle ring is pushed down to the bottom piece of the cassette. Any gap in between will allow the filter to move, and could possibly cause the collected PM cake to fall off, especially when a back pressure is created by detaching the sampling cassettes. Note that the ring does not usually sit firmly against the filter. Use a light soft-faced hammer or mallet to tap the middle ring until it sits closely on the bottom of the cassette.

- 5.6. When using desiccant dryer, minimize the time the filter/cassette is exposed to room air before and after weighing. The collected PM is usually very hygroscopic. Minimize the times the desiccant cover is removed to reduce the amount of moisture introduced into the desiccant drier. This is especially important when the humidity of room air is not controlled.
- 5.7. Transport the capped filter cassettes to the laboratory in an upright position for conditioning and weighing. Use the entire filter cassette (including the top cap) during storage and transportation, to protect the filter from contamination.
- 5.8. Remove the top piece of the filter cassette when conditioning the filters, to accelerate the conditioning process. The PM collected onto the ring and bottom piece does not usually attach to the top piece, especially when a nozzle is used; the PM mainly collects onto the lower part of the ring and the filter.
- 5.9. Before or after assembly of the filter cassette components, label each piece (except the filter) so that it is clear which top, ring, and bottom piece go with which "assembly". This will make it easier to track possible contamination later. An example would be labeling the top, ring, and bottom piece as 01T, 01R, and 01B respectively.
- 5.10. Previous studies have not accounted for sampling nozzle-deposited PM. An analysis was conducted to determine the importance of collecting PM from cleaning the heads (Lim et al., 2005). It was found that the PM deposited on the nozzle contained 15% of the total PM mass collected. It is therefore very important to recover the nozzle-deposited PM by rinsing the nozzle with acetone and determining the rinsate mass.

6. Interferences

- 6.1. Positioning any large object within 0.3 m of the sampling system can affect the airflow near the sampling nozzle.
- 6.2. The activity involved in maintaining the system, or other equipment around the sampling area, can induce more PM (for example, by stirring up dust on surfaces), thus affecting the measurements.
- 6.3. Excessive amounts of PM collected on the filters may reduce the sampling flow rate.
- 6.4. Continuous operation of the fan may be interrupted by erroneous ventilation stage control or fan failure. Monitor and ensure continuous fan operation, using a device such as vibration sensor (SOP A7) or vane anemometer (SOP A3).

7. Personnel Qualifications

- 7.1. Personnel must read this SOP and be trained in the use of the sampling system before initiating the procedures.

- 7.2. In addition, the operator must be trained to conduct leak check, flow rate, and air velocity measurement.

8. Equipment and Supplies

8.1. **Table 1. List of parts for one TSP sampling system for 2 m/s sampling velocity.**

Description	Quantity	Supplier	Catalog/ Part Number
14.6 mm aluminum isokinetic sampling nozzle - 2 m/s ($\pm 5\%$)	3	CCC Technologies	IKS-2.0-AL
20 L/min ($\pm 5\%$) critical venturi	3	CCC Technologies	V-20-BN
Monitor case, empty, transparent, 3 piece compression fir, with end caps, 50/Pk	1 pkg	Fisher Scientific	M000037A0
Whatman binder-free glass micro-fiber filters, 37-mm, 100/Pk	3 pkg	Fisher Scientific	09-874-12B
Tubing adapter, male Luer, 10/Pk	12	Fisher Scientific	XX6200005
1/3-HP gas vacuum pump	2	Grainger	4F740
Commercial vacuum gauge, dial size 2.5", NPT 1/4" lower connection, 30-0"Hg	2	Grainger	2C537
Female tee, Size 1/4", brass pipe fitting, 10/Pk	2	Grainger	6MN49
Street elbow, Size 1/4", brass pipe fitting, 10/Pk	2	Grainger	6MN80
Hex nipple, Size 1/4", brass pipe fitting, 10/Pk	4	Grainger	6MN97
3/8" brass hose barb to 1/4" NPT male	2	Grainger	4A487
Masterklee general purpose clear PVC tubing 3/8" ID, 5/8" OD, 1/8" wall thickness	50 ft	McMaster Carr	5233K65
Masterklee general purpose clear PVC tubing 1/4" ID, 7/16" OD, 3/32" wall thickness	15 ft	McMaster Carr	5233K57
Nylon multi-barbed tube fitting reducing coupling for 3/8" x 1/4" ID tube	10	McMaster Carr	5372K517
Nylon multi-barbed tube fitting 90° elbow for 3/8" ID tube	6	McMaster Carr	5372K372
Nylon multi-barbed tube fitting tee for 3/8" ID tube	10	McMaster Carr	5372K615

9. Procedures

9.1. Sampling system installation

9.1.1. General information

- 9.1.1.1. Conduct a practice assembly in the laboratory to make sure all necessary parts and tools are gathered before proceeding to the field.
- 9.1.1.2. There are three assemblies, as follows:

- 9.1.1.2.1. The filter cassette assembly consists of only the clear plastic filter cassette (3 pieces and caps) and the filter.
 - 9.1.1.2.2. The sampling nozzle-filter-venturi assembly contains the sampling nozzle, partial filter assembly, male Luer adapter, 1/4" ID PVC tube, reducing coupling for 3/8" x 1/4" ID tube, 3/8" ID PVC tube, and critical venturi.
 - 9.1.1.2.3. The TSP sampling system assembly includes the sampling head-filter assembly, the vacuum pump, and necessary fittings and tubes.
- 9.1.2. Filter cassette assembly
- 9.1.2.1. Figure 1 shows the filter cassette fitted with a glass fiber filter. The filter cassette is made of clear plastic, and consists of three main parts: the top, ring and bottom. In addition, there are two caps that seal the top and bottom of the cassette, for a total of five pieces. The bottom part is where the filter sits. The ring holds the filter down and the top and caps protect the filter during handling.

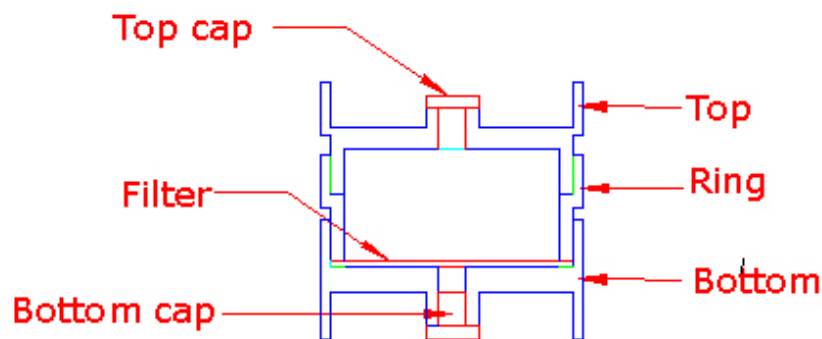


Figure 1. Filter cassette assembly.

- 9.1.2.2. Filter assembly procedure
 - 9.1.2.2.1. Use tweezers, or wear clean powder-less gloves, when handling the filters.
 - 9.1.2.2.2. Place the filter inside the bottom piece of filter cassette during assembly, with the smoother side facing down.
 - 9.1.2.2.3. Place a ring inside the bottom piece and push down so that the two pieces are held tightly together. Make sure the filter covers the entire bottom cassette and is pressed down tightly. Use a light, soft-faced hammer to carefully tap the ring and bottom cassette together if needed.
 - 9.1.2.2.4. Attach the top piece and the top and bottom caps to the assembly.
 - 9.1.2.2.5. Label every piece of the cassette (except the filter) so that it is clear which top, bottom, etc. go with which "assembly".
- 9.1.3. Figure 2 shows the sampling nozzle-filter assembly. This section consists of the sampling nozzle, a partial filter cassette assembly, the critical venturi and the necessary tubing and fittings.
 - 9.1.3.1. Connecting the sampling head-filter-venturi system

- 9.1.3.1.1. First, assemble the section up to the filter cassette (from the right) when setting up the initial system. The assembly should resemble Figure 2.
- 9.1.3.1.2. The Luer adapter is a fitting that fits inside the bottom of the filter cassette and connects it to 1/4" ID tubing.
- 9.1.3.1.3. Transition the 1/4" tubing to 3/8" ID tubing, using the provided adapters, so that the critical venturi can be inserted.
- 9.1.3.1.4. Verify that there is a minimum of one inch between the venturi and any other fittings to allow for proper air flow development.
- 9.1.3.1.5. The section described thus far will be a relatively permanent installation. Once installed, there will be minimal need to change it.
- 9.1.3.1.6. If using an isokinetic sampling nozzle, remove the top piece of the cassette, insert the nozzle into the ring (similar to the way the top was) so that it fits snugly.
- 9.1.3.1.7. Place the filter cassette bottom onto the Luer adapter.

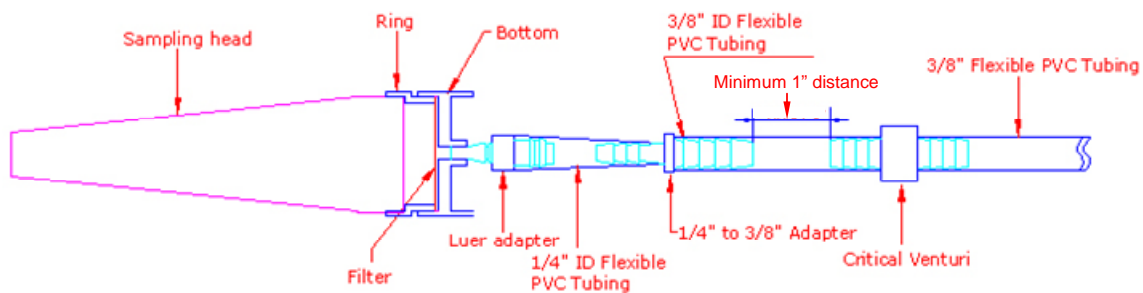


Figure 2. Sampling head–filter-venturi assembly, there are three of such assembly in a complete TSP sampling set.

- 9.1.4. Figure 3 shows the complete TSP system assembly, in which the three sampling filter assemblies are supported by utility clamps that are attached to a stand, and the vacuum pump is stored in an enclosure.
 - 9.1.4.1. The tubing is likely to be longer and the heads further apart or closer, depending on the needs of each individual user and sampling location. The heights of the filter assemblies can be easily adjusted. In some cases it may be necessary to suspend the stand from the ceiling.
- 9.2. Location of the Sampling Head
 - 9.2.1. Determine the locations and orientations of the sampling heads according to the desired local air velocity. The local air velocity refers to the face velocity at the sampling head. It is typically 2 m/s.
 - 9.2.1.1. Measure the local air velocity and choose a plane where the velocity equals the designed isokinetic sampling velocity of the head, within 10%. Generally, this velocity should be found within two diameters of the fan.



Figure 3. A TSP sampling system (three filter cassettes, tubing, and the white sampling pump box on the left) with sampling filters and tubes attached to a sturdy 2x4 post.

- 9.2.1.2. Locate the three sampling nozzles so that they are equally distributed along the height of the sampling fan. All heads do not have to be on the same plane, but they should all be on the same velocity contour to ensure isokinetic sampling.
 - 9.2.1.3. Locate the nozzles facing the incoming air stream to the fan (facing upstream away from the fan itself). If the direction of the air stream deviates significantly from perpendicular to the fan, then angle the nozzles to face into the air stream.
 - 9.2.1.4. Use a recently calibrated air velocity measurement device, such as portable vane thermo anemometer or a hot-wire anemometer, to measure air velocities when determining appropriate sampling locations.
 - 9.2.1.5. Secure sampling system so that it is not easily moved, and clearly mark its proper location.
 - 9.2.1.6. Record distances to the fan and air velocities measured for each nozzle with the sampler running.
- 9.3. System Leak and Flow Rate Check
- 9.3.1. System Leak Check
 - 9.3.1.1. Perform a leak check after initial installation, and after any significant changes to the system. It is not necessary to conduct a leak check every sampling event, especially when the individual nozzle flow rates are measured at the beginning and ending of each sampling.

- 9.3.1.2. Install the entire filter cassette assembly (excluding the bottom cap) to conduct a leak check, with the top cap in place. This should seal the system.
- 9.3.1.3. Install a flow measurement device (preferably a rotameter) at the vacuum pump outlet. The measurement range of the device should be less than 1 L/min. Turn the pump on and verify that the vacuum gage reads over 25 inches of Hg, and that the flow measurement device reads less than 1 L/min.
- 9.3.1.4. If these conditions are not met, check all connections and repeat leak check.
- 9.3.2. Venturi Flow Rate Check
 - 9.3.2.1. Use this procedure primarily as a check, not a calibration.
 - 9.3.2.2. Set up the system in a similar manner as when the system is sampling, but replace the filter cassette top of one sampling nozzle with a flow measurement device.
 - 9.3.2.3. Turn on the pump. Take at least three flow readings, and average them.
 - 9.3.2.4. Verify that this average is within 5% of the calibrated flow rate (the stated venturi flow).
 - 9.3.2.4.1. Check flow meter, leakage, and system setup if flow rate is not within 5% of the calibrated value.
 - 9.3.2.5. Turn off pump, remove flow measurement device, and replace sample nozzle cassette top.
 - 9.3.2.6. Repeat steps 9.3.2.2 – 9.3.2.5 for the other two sampling nozzles.
 - 9.3.2.7. The actual flow rates of the three nozzles will likely vary by approximately 3-5%, due to slight differences in the filters and tubing.
- 9.4. Filter preparation and weighing
 - 9.4.1. Filter handling
 - 9.4.1.1. Inspect each filter for pinholes, particles, and other imperfections.
 - 9.4.1.2. Be careful to avoid damage of the filter during handling.
 - 9.4.1.3. Handle the filters and cassettes using appropriate tweezers and gloved (clean and powder-less) hands.
 - 9.4.1.4. Place each filter in a uniquely labeled filter cassette before weighing it. Each piece of the filter cassette should be labeled. The filter should remain in this cassette until all conditioning, sampling and weighing has been completed.
 - 9.4.1.5. Archive the filters and cassettes for at least two more sampling rounds in case further analysis is necessary.
 - 9.4.2. Filter conditioning
 - 9.4.2.1. Condition labeled cassettes (with filters) in an environment-controlled room or desiccant drier for at least 24 h before weighing. The environment-controlled room should have temperature and relative humidity (RH) maintained at $25 \pm 5.6^\circ\text{C}$ and $< 50\%$ RH. RH of the room must be constant within $\pm 5\%$ RH.
 - 9.4.2.2. Weigh each filter in its own cassette with only the bottom cassette and middle ring attached, the top piece is removed.
 - 9.4.2.3. During conditioning and weighing, remove bottom cap to allow faster drying and higher weighing accuracy.
 - 9.4.3. Filter weighing
 - 9.4.3.1. Use a scale with a precision of 0.1 mg or less. The average mass of clean and loaded filters with rings and bottom cassettes ranges from 12 to 14 g, while the

filter should collect about 0.115 g of PM during a 48-h sampling period (assuming 20 L/min sampling rate and a TSP concentration of 2000 $\mu\text{g}/\text{m}^3$).

- 9.4.3.2. Close the scale door before taking any readings, and make sure the scale is zeroed before the cassette is put on.
- 9.4.3.3. Record the mass to the nearest 0.1 mg.
- 9.4.3.4. Mass should be taken daily until the mass stabilizes, when two consecutive masses differ by less than 1.5 mg (approximately 1.3% of the PM mass).

9.5. Sampling procedures

9.5.1. Start the sampling as follows:

- 9.5.1.1. Remove the tops of the pre-conditioned and labeled filter-cassette assemblies.
- 9.5.1.2. If using isokinetic heads, attach cleaned sampling heads to the rings, and check to make sure they are properly attached.
- 9.5.1.3. Insert the Luer adapters into the bottom cassettes.
- 9.5.1.4. Secure all three assemblies so that they are fixed at the correct locations and in the correct orientations.
- 9.5.1.5. Check to make sure the system is complete, and that the heads are not likely to move during sampling.
- 9.5.1.6. Program the timer (if using one) for the required sampling duration (multiple of 24 h) as listed in the QAPP.
- 9.5.1.7. Turn on the sampling system.
- 9.5.1.8. Record sampling details, including barn number, cassette assembly numbers for each of the top, middle, or bottom sampling positions, and sampling starting date and time.
- 9.5.1.9. Attach a flow measurement device onto each sampling head or filter-cassette assembly to measure the sampling flow rate. Record the values.
- 9.5.1.10. If using isokinetic sampling heads, conduct an air velocity check. Verify that velocities are within 10% of the design velocity. Record the air velocities and adjust the sampling locations if needed.

9.5.2. At the conclusion of sampling:

- 9.5.2.1. Measure and record sampling flow rates again before shutting system off.
- 9.5.2.2. Remove filter-cassette assemblies from the system. Be careful when handling filter-cassettes. Keep assembly upright after it has been exposed. There will likely be a significant PM cake, which can easily fall off if the filter is turned over or shaken.
- 9.5.2.3. Attach the matching labeled top pieces to the rings. If isokinetic heads are used, remove them without removing the filter cassette rings before attaching the top cassette covers. Rinse the sampling heads with acetone (See Section 9.6) to collect PM which has been deposited on the heads themselves.

9.6. Recovering sampling head-deposited PM

- 9.6.1. This section applies only to the use of isokinetic sampling heads, and should be skipped if the TSP sampling is conducted without a sampling head (e.g. for still air sampling).
- 9.6.2. After capping the filter-cassette, individually rinse the inside surfaces of each sampling head with acetone. The amount of acetone used should be 25 to 100 mL.

- 9.6.3. Brush the inside surfaces with a nylon bristle brush while rinsing. The brush does not usually collect significant amount of PM. However, always spray clean the brush with acetone and visually inspect the brush between nozzle cleaning.
- 9.6.4. Collect all acetone rinsate in 250-mL glass bottles, and transport to the laboratory to determine PM mass in the rinsate.
- 9.6.5. Evaporate the rinsate at room temperature in aluminum weighing dishes, and condition and weigh them as specified in section 9.4. Add rinsate PM mass to the TSP collected on the filters to calculate PM concentrations.
- 9.7. Troubleshooting
 - 9.7.1. If the flow check is out of specification:
 - 9.7.1.1. Check vacuum pump.
 - 9.7.1.2. Check for leakage in the lines, and in the connections of each element.
- 9.8. Data acquisition, calculations & data reduction requirements
 - 9.8.1. Validation of samples using field blank (FB) filters. Use the FB filters to determine if there is any problem with the filter handling procedures. The validation procedure uses the average of the applicable blanks (B_{AVG}) and the standard deviation of the blanks (B_{σ}). If the sample mass (M_t , of filter loaded with PM) satisfies: $M_t > B_{AVG} + 5 \times B_{\sigma}$, Then the sample is valid (McClure et al., 2004).
 - 9.8.2. Concentration and emission calculations
 - 9.8.2.1. Calculate the total air flow of each sampling line based on sampling flow rates and recorded sampling duration. The sampling flow rate is the average of the beginning and final rate of each sampling event.
 - 9.8.2.2. Calculate the concentration by combining the mass of TSP collected in the acetone rinse and on the conditioned filters, then dividing the combined mass by the total sample air volume.
 - 9.8.2.3. Calculate and report the emission rates according to SOP B4.

10. Data and Records Management

- 10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet that is designated for this method. Supplement this electronic record with a bound record book designated for the method, which should also contain bound copies of the electronic record.
- 10.2. Manage data according to SOP B5.
- 10.3. Document all data and information on field data sheets, and site logbooks with permanent ink, or in electronic field notes.
- 10.4. Maintain a Filter Identification Record for each assembly, tracking each use (time and location), weights, and results of all leak and flow checks.
- 10.5. Use Chain of Custody forms when shipping filters.

11. Quality Control and Quality Assurance

- 11.1. Venturi calibration. A certification of calibration can be obtained from the manufacturer (UIUC lab).

- 11.2. Conduct field blank samples (the numbers and frequency of which will be specified by the QAPP) in the same manner as the normal TSP sampling, but remove the assemblies before the pump is turned on, to avoid exposure to barn airflow.
- 11.3. Treat blank samples exactly as if they were normal loaded filters for conditioning and weighing.
- 11.4. Recalibrate or replace venturi when the flow rate is not within 5% of the calibrated flow rate.
- 11.5. Analytical balance calibration and standardization
 - 11.5.1. Calibrate the balance annually (at a minimum).
 - 11.5.1.1. Calibrate at 2 mg and 1000 mg using class "S" weights.
 - 11.5.1.2. Instrument calibration must be $< \pm 10\%$ at 2 mg (i.e. ± 0.2 mg) and $\pm 0.5\%$ at 1000 mg (i.e. ± 5 mg). If these limits are not achieved, recalibrate the balance.
 - 11.5.1.3. Place a 50-g weight and the 2-mg weight on the balance together. Verify that the balance reads 50.002 g (± 0.0002 g).

12. References

- 12.1. Lim, T.T., H.W. Sun, J.-Q. Ni, L. Zhao, C.A. Diehl, A.J. Heber, and P.-C. Tao. 2005. Field tests of a particulate impaction curtain on emissions from a high-rise layer barn. In Proceedings of ASAE Annual International Meeting. ASAE, St. Joseph, MI.
- 12.2. McClure, J.; Jerez, S.; Zhang, Y. 2004. An Isokinetic Total Suspended Particulate sampler for measurement of emissions from confinement animal buildings. In Proceedings of the 97th Annual Meeting & Exhibition of American Waste Management Association. AWMA: Pittsburgh, PA.
- 12.3. Wang, X.; Zhang, Y. 1999. Development of a critical airflow venturi for air sampling. *Journal of Agricultural Engineering Research* 73: 257-264.
- 12.4. Jerez, S.B, Y. Zhang, J. McClure, L. Jacobson, A. Heber, S. Hoff, J. Koziel, and D.B. Beasley. 2006. Comparison of measured total suspended particulate matter concentration using tapered element oscillating microbalance and a TSP sampler. *J. Air & Waste Management Association*. 56:261-270.
- 12.5. SOP A3. 2006. Exhaust Fan Air Velocity Measurements Using Vane Anemometry. Standard Operating Procedure A3. Purdue Ag Air Quality Lab.
- 12.6. SOP A7. 2006. Fan Status Monitoring Using Vibration Sensors. Standard Operating Procedure A7. Purdue Ag Air Quality Lab.
- 12.7. SOP B4. 2006. Calculation and Reporting of Air Emissions from Barns. Standard Operating Procedure B4. Purdue Ag Air Quality Lab.
- 12.8. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.

13. Manufacturer's Contact information

- 13.1. Fisher Scientific, 2000 Park Lane, Pittsburgh, PA 15275
- 13.2. Grainger, 100 Grainger Parkway, Lake Forest, IL 60045
- 13.3. McMaster Carr, 600 County Line Road, Elmhurst, IL 60126
- 13.4. PAAQL: odor@purdue.edu, heber@purdue.edu, <http://www.AgAirQuality.com>

**MEASUREMENT OF PM_{2.5} WITH
PARTISOL-FRM MODEL 2000 AIR SAMPLER**

Standard Operating Procedure (SOP) P3

**MEASUREMENT OF PM_{2.5} WITH PARTISOL-FRM
MODEL 2000 AIR SAMPLER
Standard Operating Procedure (SOP) P3**

Prepared by

Juan Carlos Ramirez and Teng-Teoh Lim

Reviewed by

Bill W. Bogan and Albert J. Heber

Effective Date: November 6, 2006

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability.....	3
2. Summary of Method.....	3
3. Definitions & Symbols.....	5
4. Health and Safety.....	6
5. Cautions.....	6
6. Interferences.....	6
7. Personnel Qualifications.....	7
8. Equipment and Supplies.....	7
9. Procedures.....	9
10. Data and Records Management.....	29
11. Quality Control and Quality Assurance.....	29
12. References.....	29
13. Contact Information.....	30
Appendix A. Measurement Quality Objectives for FRM PM _{2.5}	31
Appendix B. Quality Control Maintenance and Calibration Schedule.....	32
Appendix C. Monthly Quality Control Maintenance Check Sheet.....	33
Appendix D. Filter Field Sample Report and Chain-of-Custody Form.....	34
Appendix E. Sampler Calibration Datasheet.....	35
Appendix F. Lab Quality Control Maintenance Schedule.....	36
Appendix G. Training Certification Evaluation Form. Field Sampling Procedures.....	37
Appendix H Training Certification Evaluation Form. Laboratory Procedures.....	40

1. Scope and Applicability

- 1.1. The TEC Partisol-FRM Model 2000 single-channel sampler and the WINS impactor will be used for integrated gravimetric sampling of particulate matter (PM) with an aerodynamic diameter less than or equal to a nominal 2.5 μm ($\text{PM}_{2.5}$) in ambient air and in exhaust air of confined animal houses or barns.
- 1.2. This procedure follows the federal reference method requirements described in 40CFR50 Appendix L.
- 1.3. $\text{PM}_{2.5}$ measurements are a 24-h integrated sample, and samples will be collected (typically) once every one or three days, depending on the PM concentration and availability of the site engineer.
- 1.4. The $\text{PM}_{2.5}$ concentration in barn exhaust air is expected to be less than 100 $\mu\text{g}/\text{m}^3$.

2. Summary of Method

The TEC Model 2000 single channel sampler is designated as a FRM (RFPS-0498-117) for collection of $\text{PM}_{2.5}$ (when using the WINS impactor). The sampler's systems perform all the functions required in the instrument specification portion of the $\text{PM}_{2.5}$ FRM standard. The federal standard requires a fixed volumetric flow rate of 16.67 L/min, equivalent to a sampling flow rate of one 1.0 m^3/h , using a specified PM_{10} inlet, tubing (downtube), secondary size-selective impactor, filter holder, and filter cassette (Fig. 1). The sampler draws ambient air through its PM_{10} inlet (Fig. 2), $\text{PM}_{2.5}$ Well Impactor Ninety-Six (WINS, Fig. 3), and a 47-mm diameter Teflon sample filter, which traps the $\text{PM}_{2.5}$ fraction. The sample filter is conditioned and weighed before and after sampling. The resulting difference in weight is the collected $\text{PM}_{2.5}$ mass in micrograms (μg). Electronic systems in the sampler are designed to monitor and maintain the volumetric flow rate as well as record the elapsed sampling time enabling the TEC Partisol-FRM Model 2000 to calculate the total sample volume in cubic meters (m^3). With this information, the analyzing laboratory will calculate and report the average $\text{PM}_{2.5}$ concentration for the sampling period in $\mu\text{g}/\text{m}^3$. The TEC single-channel sampler monitors and regulates the flow rate using the sampler's microprocessor, software, mass flow controller, ambient temperature sensor, and ambient pressure sensor. The sampling period can be between one to three days (24 to 72 h). The flow rate of the sampler must be 16.67 L/min (5-min average over a 24-hr period) $\pm 5\%$. For a more detailed explanation of the sampler's theory of operation, please read Section 1: Introduction, of the Operating Manual (2004, version B) and see Figs. 1 through 3 below.

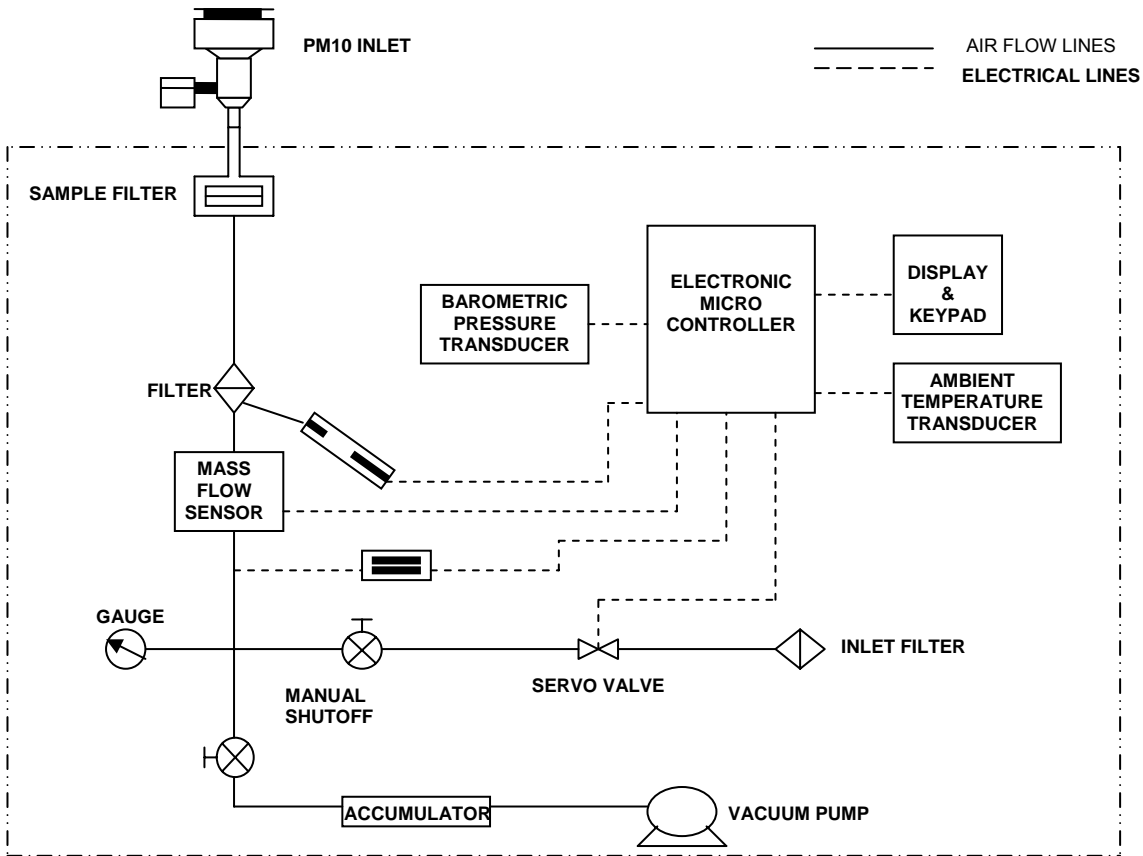


Figure 1. Schematic of TEC Model 2000 PM_{2.5} Single Channel Air Sampler.

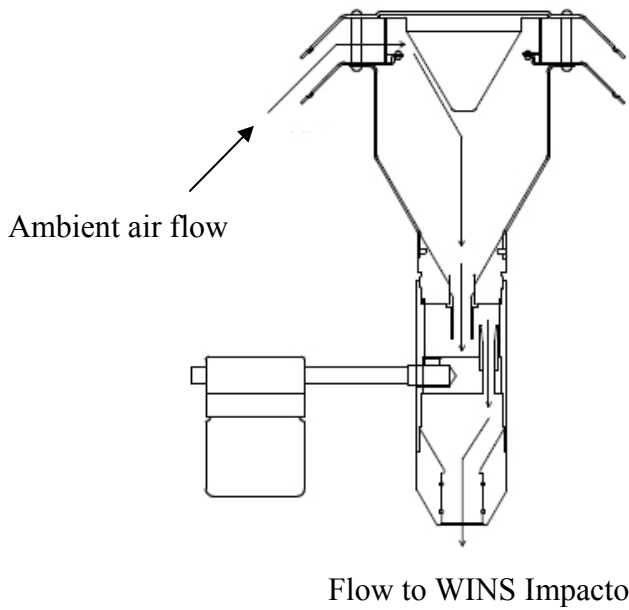


Figure 2. Air flow through the PM_{2.5} sampler impactor.

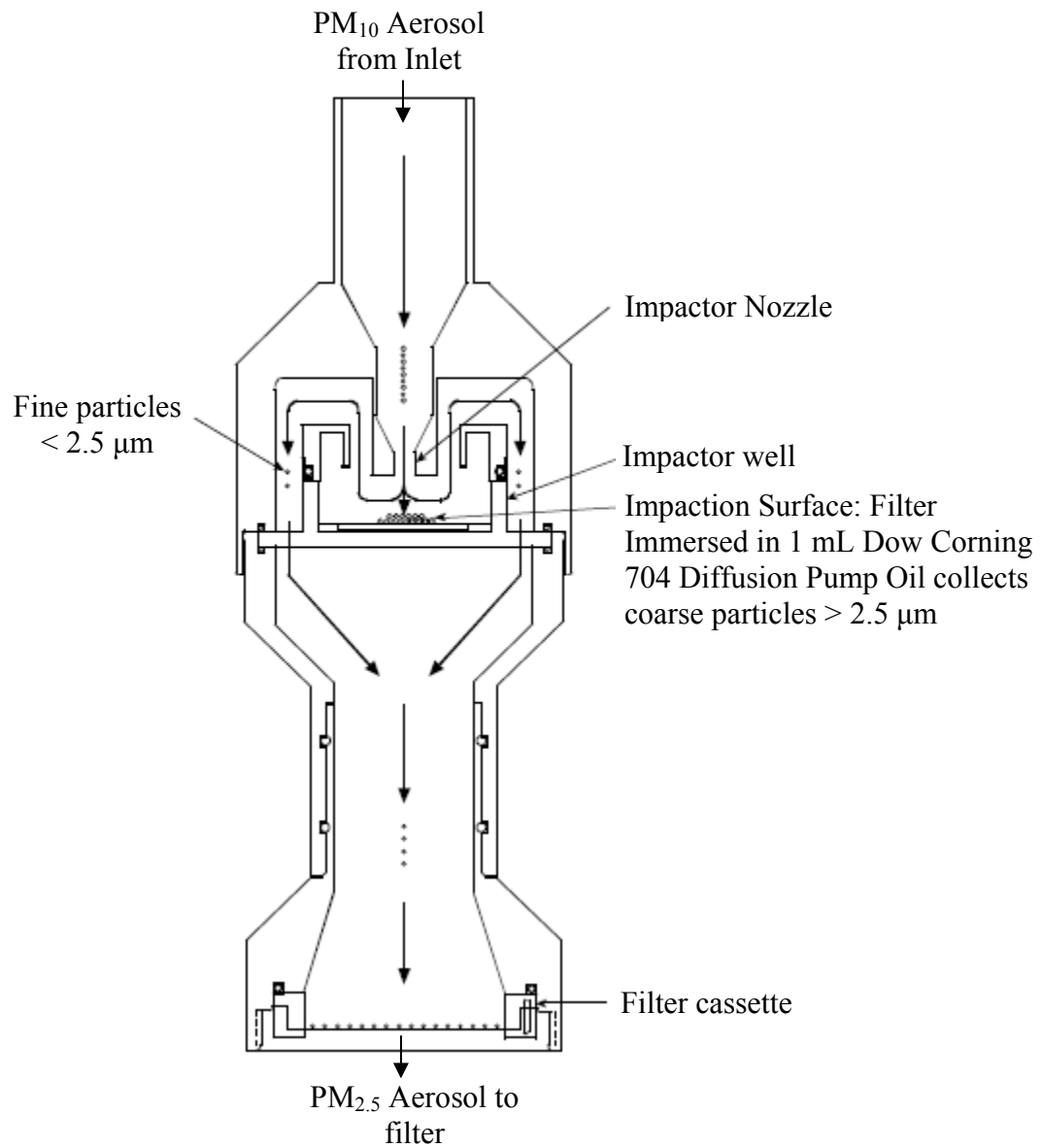


Figure 3. WINS impactor, to separate PM_{2.5} from PM₁₀.

3. Definitions & Symbols

- | | | |
|------|------|---|
| 3.1. | A/I | Analog input |
| 3.2. | DVM | Digital volt meter |
| 3.3. | FRM | Federal Reference Method |
| 3.4. | FRS | Flow rate standard |
| 3.5. | FTS | Flow transfer standard |
| 3.6. | GND | Ground connection located on the electronic board of the sampler. |
| 3.7. | NIST | National Institute of Standards and Technology |
| 3.8. | PFM | Primary flow meter |

- 3.9. PM Particulate matter
- 3.10. PM_{2.5} Particulate matter with an aerodynamic diameter less than or equal to a nominal 2.5 µm
- 3.11. QC Quality control
- 3.12. TEC Thermo Electron Corporation
- 3.13. VAC Voltage alternating current
- 3.14. WINS Well Impactor Ninety-Six (separator used in the FRM for PM_{2.5})
- 3.15. <...> Indicate a command on the screen of the instrument.

4. Health and Safety

- 4.1. High voltages (120 VAC) are used to power the unit.
- 4.2. When assembling some components (e.g. the WINS impactor) take care not to pinch fingers between assembly parts.
- 4.3. Do not use this instrument outdoors without the proper exterior power connection, or in ambient temperatures outside the range of -40° to 50°C (-40° to 122 °F).
- 4.4. Unplug the sampler whenever possible while working around electrical components. Working outdoors in ambient (wet) weather conditions increases the risk of electrocution.
- 4.5. Consult the Material Safety Datasheets (MSDS) for safety information about silicon oil, halocarbon grease, and other materials that may be used to clean the TEC sampler parts.
- 4.6. For additional safety information, read Section 2.2.5: Hardware Installation and Maintenance of the TEC Operating Manual (2004).

5. Cautions

- 5.1. Installation, operation, maintenance, and calibration of the sampler should only be performed by properly trained personnel.
- 5.2. Temperature and relative humidity must be controlled during weighing and transporting of the filters according to the specifications for this method.
- 5.3. Do not calibrate the instrument's flow rate if the temperature and pressure sensor have not been calibrated first.
- 5.4. Use proper anti-static protection when the door to the electronics compartment is open and when taking measurements on any electronic components. Failure to use anti-static protection could result in damage to electronic components.

6. Interferences

- 6.1. Variations in flow rate limit the precision for sampling PM_{2.5}. The calibration of the flow rate is of high importance.
- 6.2. Weight loss due to mechanical removal of particles from the filter is minimized by carefully removing the filter, in its cassette, from the sampler and storing the filter/cassette in a protective container during transit to the weighing laboratory.
- 6.3. Filter weight loss due to outgassing can be minimized by allowing a six-week period before the filter is used (EPA Quality assurance Document 2.12, Section 7.6, page 7 of

- 19). Conduct this equilibration period in the same conditioning room, previous to the initial weighing of the filter.
- 6.4. Weight loss can also be minimized by carefully removing the filter from the cassette, conditioning the filter, and neutralizing the static charge on the filter before weighing.
 - 6.5. Errors in the gravimetric analysis of samples can also result from the buildup of electrostatic charges on filters during their manufacture or during sampling. This static buildup will interfere with microbalance weighing, but can be reduced or eliminated by using Polonium-210 (^{210}Po) antistatic strips before beginning the weighing process.
 - 6.6. If nitric acid vapor is present at a sampling location, it can deposit on a Teflon[®] filter and cause a small weight gain in proportion to the concentration of nitric acid in the atmosphere. This weight gain may not be controllable.

7. Personnel Qualifications

- 7.1. The operator of this procedure must be trained in advance, under supervision, before he or she can perform it alone. Analysts must practice filter loading and unloading before performing the procedure with an actual test filter.

8. Equipment and Supplies

- 8.1. Ensure that each sampler has the supplies included in Table 1.

Table 1. Supplies for the TEC Partisol-FRM Model 2000 PM_{2.5} Air Sampler.

Quantity	Part Description
1	Partisol-FRM enclosure with WINS PM _{2.5} impactor and filter exchange mechanism.
1	Electronic max-min thermometer (to be used in the filter transport container)
1	1 st stage PM ₁₀ inlet
1	Sample tube
1	Flow audit adapter
1	Dual filter transport container with cassettes and carriers
1	Ambient temperature sensor and cable
3	Set of impactor wells and anti-spill rings
4	Set of filter cassettes and backing screens
2	Set of inlet O-rings
1	Bottle (100 mL) of WINS impactor oil
1	Box (50 count) of glass fiber impactor filters, 37 mm
1	Analog input calibration cable
1	Mating cable connector for four-pin user output connector
1	AKCOMM software diskette
1	9-to-9 pin computer cable
2	Operating manuals
1	Service manual
1	Quick Start Guide

- 8.2. Upon receipt of the sampler(s), inspect sampler and accessories for shortage and for shipping damage. If shortage or damage is found, immediately notify the site's project manager.
- 8.3. Keep the following consumables (Table 2), Sampler accessories (Table 3) and batteries (Table 4) on hand with each sampler (i.e. on-site). Part number refers to TEC number.

Table 2 Consumables for the TEC Partisol-FRM Model 2000 PM_{2.5} Air Sampler.

Part Description	Part Number
Polonium-210 (²¹⁰ Po) antistatic strips	N/A
Teflon filters, 2.0 µm pore size, box of 50	10-002322-0050
Single filter carrier	55-004847
Dual-filter transport container	20-004996
Partisol-FRM filter cassette for 47-mm filters	59-004648-0001
5 Partisol-FRM filter cassettes for 47-mm filters	59-004648-0005
10 Partisol-FRM filter cassettes for 47-mm filters	59-004648-0010
Filter cassette screens, package of 5	30-005147-0005
Solid filter leak check/separator disk	36-004768
Tweezers	30-002566
Filter cassette separator tool	38-004892

Table 3 Accessories for the TEC Partisol-FRM Model 2000 PM_{2.5} Air Sampler

Part Description	Part Number
WINS PM _{2.5} impactor	57-004006
Impactor assembly	55-004289
WINS O-ring, 2 3/8" ID x 2 1/2" OD x 1/16", BUNA	22-0002853-3036
Box of 25 glass fiber filters, 37 mm	32-004294
Bottle of WINS impactor oil, 30 ml	59-004292
Bottle of WINS impactor oil, 100 ml	59-004792-0100
PM ₁₀ inlet	57-004742
PM ₁₀ inlet O-ring 1 1/4" ID x 1 3/8" OD x 1/16", BUNA	22-00485-1026
PM ₁₀ inlet O-ring 2 3/8" ID x 2 1/2" OD x 1/16", BUNA	22-000485-1036
Large in-line filters located in the pump compartment (Change every 6 m.)	32-002643

Table 4 Batteries for the TEC Partisol-FRM Model 2000 PM_{2.5} Air Sampler

Part description	Part Number
Round battery for computer board (25 mm dia, Panasonic CR2330)	06-002545
Battery for socket at location U4 (Dallas Semiconductor DS1213C)	06-002565

- 8.4. Aside from the 47-mm collection filters, the 37-mm WINS impactor glass fiber filters, the impactor oil, and the batteries, the only regular “consumable” item in Tables 1-4 is the large in-line filter located in the pump compartment. This item should be exchanged every six months, as noted in Table 3.
- 8.5. The equipment necessary to conduct a sampler calibration is presented in Table 5.

Table 5 Apparatus for Partisol-FRM single-Channel Sampler Calibration.

Part description
Certified flow rate standard
Certified reference temperature meter
Certified pressure meter
TEC inlet flow audit adapter
Tubing (Tygon or surgical type)
Blank filter
Calibration forms
Digital Voltmeter (DVM)
Container for temperature calibration
Flow-check filter in transport cassette
Impermeable “filter” disk for internal leak checks
Accurately set timepiece
Hand calculator

9. Procedures

- 9.1. Equipment and supplies inspection
- 9.1.1. Table 6 shows acceptance and limits for procurement of equipment and supplies.
- 9.2. Installation
- 9.2.1. Physical inspection
- 9.2.1.1. Upon receipt, inspect sampler and accessories for shortage, and for shipping damage. If shortage or damage is found, immediately notify the site Principal Investigator and/or PAAQL personnel.
- 9.2.2. Initial sampler installation: Follow directions found in Section 2 of the TEC Operating Manual for installation instructions, and consult with the site Principal Investigator to assure that the installation site complies with the project’s siting criteria for FRM PM_{2.5}.

Table 6. Acceptance checks and limits for procurement of equipment and supplies.

Equipment	Acceptance check	Acceptance limits	Action if requirements are not met
Field operations			
Sampler	Sampler and accessories complete; no evidence of damage. Model designated as reference or equivalent method. Pump and display work.	Type as described in Appendix A	Reject sampler
Calibration quality assurance/quality control (QA/QC) equipment for flow rate, temperature, pressure.	Accompanied by certificate. Check values against National Institute of Standards and Technology (NIST)-traceable standards.	Within accuracy limits in Appendix A	Adjust or reject equipment
Audit equipment	Same as for calibration equipment but must not be the same equipment.	Type as described in Appendix A	Adjust or reject equipment
Laboratory operations			
Filters, Teflon®	Of correct type and undamaged.	Type as described in Table 7	Reject filters
Filter cassettes	Of correct type and undamaged.	As specified by sampler manufacturer	Reject filter cassettes
Filter/cassette protective containers	Of correct type and undamaged.	As specified by sampler manufacturer	Reject containers
Filter-handling containers	Of correct type and undamaged.	As specified by sampler manufacturer	Reject containers
Analytical microbalance	Accompanied by certificate; check values against working standards.	Readability: 1 µg, Repeatability: 1 µg	Adjust or reject equipment
Mass reference standards	Check working standards against NIST-traceable primary standards.	Tolerance: ±25 µg from nominal value	Reject standards.

Table 7. Filter specifications (from 40CFR Part 50, App L).

Filter Design Specifications	Acceptance Criteria
Size	46.2 mm dia + 0.25mm
Medium	Polytetrafluoroethylene
Support ring	Polymethylpentene
	0.38 mm thick
	46.2 mm + 0.25mm outer diameter
	3.68 (+0.00, -0.51mm) width
Pore size	2 µm
Filter thickness	30-50 µm
Max. pressure drop	30 cm H ₂ O @ 16.67 L/min
Max. Moisture pickup	10 µg increase in 24 hr.
Collection efficiency	99.7%
Filter weight stability	<20 µg
Alkalinity	< 25.0 microequivalents/g

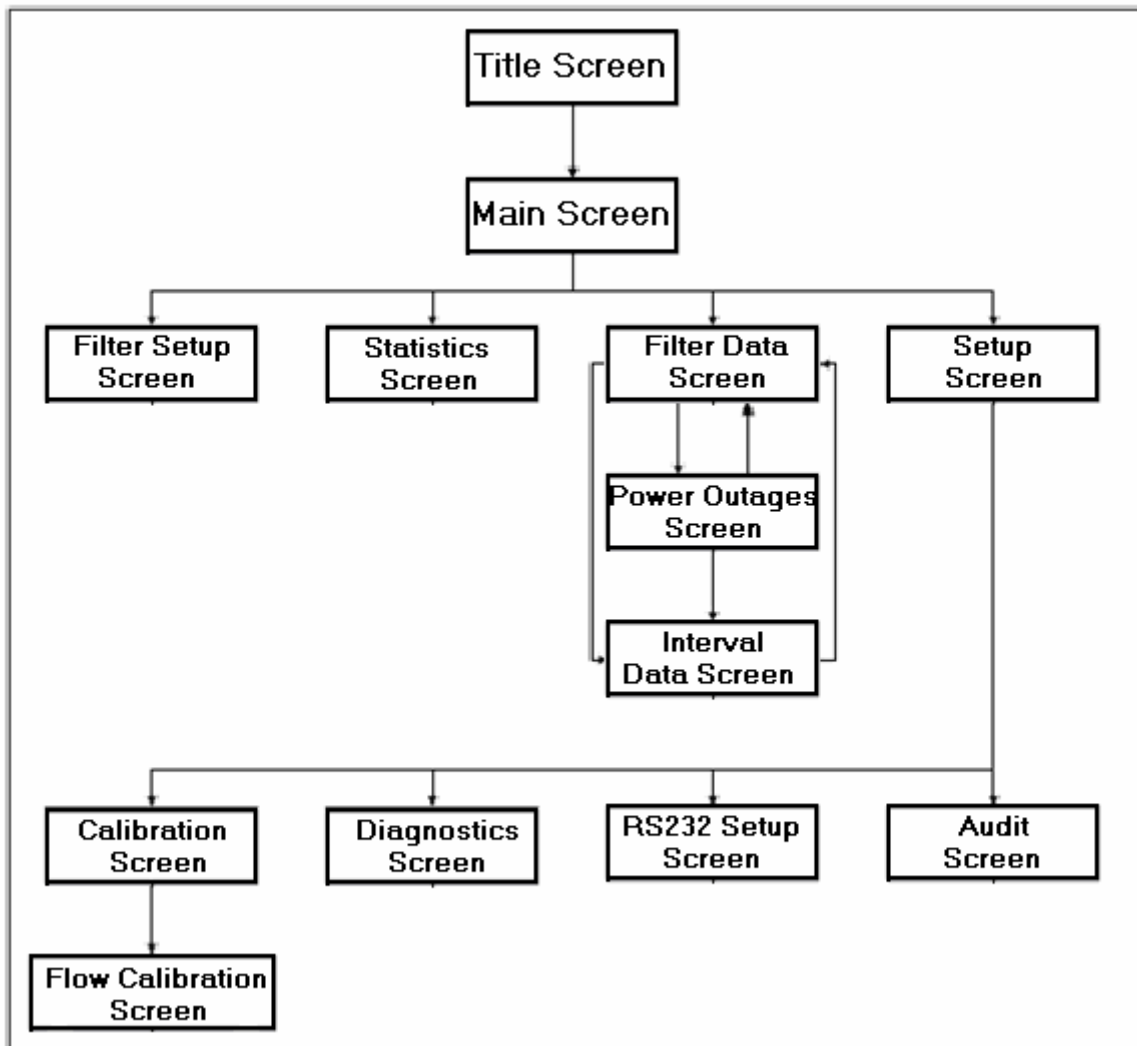
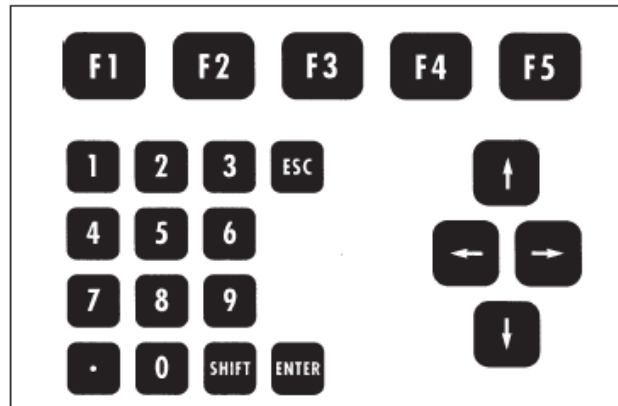


Figure 4. Hierarchy of screens on the TEC Keypad.

9.3. Initial sampler software setup: Follow directions found in Sections 4, 5, and 6 of the TEC Operating Manual.

9.3.1. Current Time: Fig. 4 presents diagrams of the TEC keypad and a hierarchy of screens which can be viewed from the sampler's display screen. From the "Main Screen", ensure the sampler is in the "STOP" operating mode. Then hit <Setup (F5)>, and cursor to the "Current Time". Hit <Edit (F1)> and set the current time.

Note: Set current time to the local standard time.

9.3.2. Start Time: Use the arrow keys to move cursor to "Start Time". Enter as 0.00.

9.3.3. Date: Use the arrow keys to move cursor to "Current Date". Enter the current date as: YY/MM/DD

9.3.4. Duration: Use the arrow keys to move cursor to the default "Duration". Set the duration to 24.00 h.

9.3.5. Flow Rate: Use the arrow keys to move cursor to "Set Flow", and set the flow rate to 16.7 L/min.

9.3.6. Serial Number: Use the arrow keys to move cursor, and enter the last 4 digits (XXXX) of the sampler's serial number into the "serial number" cell.

9.3.7. To save all setup parameters, hit the <ENTER> touch-key.

9.4. Periodic verification/checks

9.4.1. General information

9.4.1.1. QC maintenance procedures (checks) are designed to help assure that valid data is produced as a result of proper sampler operation and maintenance, in accordance with its federal designation and the manufacturers operating manual.

9.4.1.2. The maintenance frequency presented in this SOP should be considered the minimum required, even though the actual frequency of performing some of these checks may vary from site to site due to different environmental factors. These may include the sampling schedule, particulate concentrations, or seasonal factors, which may require an increase in maintenance frequency (Appendix B).

9.4.1.3. In the event that these checks cannot be performed on schedule, the deferred maintenance should be performed as soon as practical. The QC schedule for field equipment is presented in Appendix B.

9.4.1.4. When QC checks are performed, record the date, results, and any pertinent comments onto the Monthly Quality Control Maintenance Checksheet (Appendix C) for the FRM PM_{2.5} Filter Sampler (QC Checksheet) presented in Appendix A. Forward Appendix C to the site project manager on a monthly basis for subsequent review and filing. Make a copy to be kept at the field site for later reference by the operator or a site visitor.

9.4.1.5. Maintenance of the TEC sampler is discussed in Section 10 of the TEC manual.

9.4.2. After each run:

9.4.2.1. Review the summary data for reasonableness and for compliance with the Measurement Quality Objectives for FRM PM_{2.5}, (Appendix A, items listed for every 24 h or every sampling period).

9.4.2.2. If questionable summary data for flow, temperature, or pressure are seen, download the 5-min data averages using the data acquisition software, and

examine these averages for values outside of the measurement quality objectives presented on Appendix A.

- 9.4.2.2.1. The volumetric flow rate, measured or averaged over a 24-hour period, shall not vary more than $\pm 5\%$ from the specified 16.67 L/min rate over the entire sample period.
- 9.4.2.3. Procedures for downloading these data are presented in Section 9 and Appendix D of the TEC manual (2004, Rev. B).
- 9.4.2.4. In the event that anomalies are present in the 5-min averages, troubleshoot the sampler according to Section 12 of the Appendices of the TEC manual.
- 9.4.2.5. During the procedure of unloading the sample cassettes from the sampler, record the sample summary data onto the sample cassette's matching Filter Field Sample Report (Appendix D).
- 9.4.2.6. Keep the filter cassettes clean. Wipe with a clean dry cloth as required. Examine the upper and lower rubber cassette seals and replace if they are cracked or damaged. Keep the rubber seals clean. Also, visually inspect the PM₁₀ inlet's water collector jar and drain it if water is present.
- 9.4.3. After every five (5) sampling runs:
 - 9.4.3.1. Remove the WINS impactor from the sampler and inspect impaction well to determine size of particulate cone, which may have formed in the center of the well. A cone taller than 2 mm, or a cone with its top broken off, indicates a need for more frequent well cleaning to minimize the possibility of particle bounce and re-entrainment of particles larger than 2.5 μm .
 - 9.4.3.2. Clean or replace impactor well with a newly serviced well. Refer to the procedures in Appendix H.2 of the TEC manual for instructions on servicing the impactor. Record performance of this procedure and pertinent comments onto the sampler's QC Checksheet (Appendix C).
 - 9.4.3.3. After servicing the WINS impactor and reinstalling, perform a leak test and record the results on the sampler's QC Checksheet (Appendix C). If the results of the leak check do not meet the criteria, investigate and repair the source of the leak.
- 9.4.4. Monthly checks
 - 9.4.4.1. Clean the interior of the sampler chassis with a damp cloth. Remove the air intake filter and clean it with soap and water. Verify that the filter is dry before it is installed again.
 - 9.4.4.2. Clean air intake fan blades with a damp cloth or brush if necessary.
 - 9.4.4.3. Inspect the sampler o-rings and gaskets.
 - 9.4.4.4. Verify that the sampler's clock time is within 10 min of standard time, as compared to a clock standard such as the telephone service time. If the difference is > 10 min, reset the sampler's clock to within 1 min of the standard, according to instructions given in Section 4.3 of the TEC manual.
 - 9.4.4.5. Record the date that these procedures were performed and the results obtained onto the sampler's Monthly QC Checksheet (Appendix C).
 - 9.4.4.6. Perform the single-point flow check using an actual flow rate or volume measuring device having an accuracy of at least $\pm 2\%$ of full scale (0-20 L/min vol-o-flow, mass flow meter, etc.) and which is calibrated or certified annually

- against a NIST-traceable standard (Section 9.5.5.1). If the sampler's flow rate verification (at the sampler's operational flow rate) differs by $\pm 4\%$ or more from the flow rate measured by the flow rate standard, perform a new multipoint calibration and repeat the flow rate verification. If a cause for the flow discrepancy cannot be found, contact the manufacturer for assistance. Record the date that these procedures were performed and results of the single-point flow check onto the sampler's QC Checksheet (Appendix E).
- 9.4.4.7. Perform a single-point check of ambient pressure and ambient temperature sensors using a temperature and pressure standard which is calibrated or certified annually against a NIST-traceable standard. Ensure the sampler's measurements are within the acceptance criteria described in Appendix A, Monthly QC checks (± 10 mm Hg for pressure, and $\pm 2^\circ\text{C}$ for temperature).
- 9.4.4.8. Trip blanks and field blanks will be initiated by the lab. The lab will designate a sample cassette as a trip blank or a field blank.
- 9.4.4.8.1. Trip blanks
- 9.4.4.8.1.1. Ship trip blank filters in cassettes with metal covers on a monthly basis.
- 9.4.4.8.1.2. Leave the metal cassette covers on the trip blank filters
- 9.4.4.8.1.3. Leave the trip blank filters in the shipping cylinder.
- 9.4.4.8.1.4. Record the relevant trip information for the trip blank on the Chain-of-Custody section of the "Filter Field Sample Report" in Appendix D .
- 9.4.4.8.2. Field blanks
- 9.4.4.8.2.1. Ship field blanks to each site on a monthly basis.
- 9.4.4.8.2.2. Take field blanks from the shipping canister and place in the filter transport container.
- 9.4.4.8.2.3. Take the regular sampling filter and the field trip blank to the location of the sampler.
- 9.4.4.8.2.4. Remove the previously sampled filter and place in the filter transport container.
- 9.4.4.8.2.5. Load the new field blank filter into the sampler and leave it there for 1 min.
- 9.4.4.8.2.6. Remove the field blank filter and cap it with the metal covers.
- 9.4.4.8.2.7. Place the field blank filter in the filter transport container.
- 9.4.4.8.2.8. Record the date, time, and length of filter exposure on the Chain-of-Custody section of the "Filter Field Sample Report" in Appendix D, for the field blank.
- 9.4.4.8.3. Treat these samples in the same manner as a regular sample cassette used for sampling with the sole exception that it will not be used to collect a sample.
- 9.4.4.8.4. Load and unload the field blank sample cassette from the sampler, transport, store and ship as usual, but do not program the sampler for a sampling event using this cassette. Perform a leak check of the sampler to ensure that a leak has not been introduced by the previous procedures.
- 9.4.5. Every 14 sampling runs:
- 9.4.5.1. Disassemble and clean the PM₁₀ inlet, sampler downtube, and the entire WINS impactor assembly.

- 9.4.5.2. Inspect o-rings for abrasions, breaks, tears, deformations or other damage. If necessary, replace o-rings and lubricate them with a light coating of halocarbon or silicone vacuum grease prior to reassembly.
- 9.4.5.3. Using the same lubricant, also lightly lubricate any aluminum threads and take extra care that the fine threads are not cross-threaded during assembly.
- 9.4.5.4. Appendix H.1 of the TEC manual discusses the procedure for maintenance of the PM₁₀ inlet.
- 9.4.5.5. After reassembly, perform a leak check according to Sections 10.2.6 of the TEC manual and record the results on the Monthly QC Checksheet (Appendix C). Ensure the results of the leak check meet the criteria in Appendix A.
- 9.4.6. Semiannual checks (every 6 mos)
 - 9.4.6.1. Perform an as-is calibration verification check of temperature, pressure and flow.
 - 9.4.6.1.1. If any of the sampler's calibrated systems fail to meet the measurement quality objectives presented in Appendix A, calibrator must adjust the parameter to bring it within specification, and conduct a multipoint calibration according to the instrument manual.
 - 9.4.6.2. Inspect the sampler's interior, PM₁₀ inlet, and WINS impactor for cleanliness and condition.
 - 9.4.6.3. Replace the large in-line filter. Turn the sampler off for this procedure.
 - 9.4.6.4. Check the voltage level of the main computer board's battery. See Appendix F.1 of the TEC manual for details.
- 9.4.7. Measure the temperature of the station freezer daily. Post a calendar on the freezer, and record the time checked and the temperature. Initial each day's entry.
- 9.4.8. Annual checks
 - 9.4.8.1. Re-certify or re-calibrate the flow rate, temperature, and pressure QC verification check measurement standards against a NIST-traceable standard. Record the date that these procedures are performed in the sampler's QC Checksheet.
- 9.5. Calibration
 - 9.5.1. Scope of the calibration procedures
 - 9.5.1.1. The following procedures describe the techniques used to verify and calibrate the TEC Partisol-FRM Model 2000 PM_{2.5} single-channel air sampler (TEC 2000). The purpose of this written procedure is to outline the techniques that will be used to calibrate the volumetric flow of the TEC 2000 sampler at 16.7 L/min (1 m³/h) (40 CFR Part 50, Appendix L).
 - 9.5.1.2. The calibration procedures listed in the TEC manual (Section 11) are fairly straightforward with regard to the necessary information and diagrams. This procedure clarifies anomalies in the TEC manual in a step-by-step calibration format.
 - 9.5.1.3. Through the normal course of operation, circumstances may arise in which the sampler will need to be reset. If this occurs, span and offset values may need to be reentered. Keeping an accurate and handy record of the verification and calibration values (see Appendix E, TEC PM_{2.5} Partisol-FRM Single Channel Sampler Calibration Datasheet) can help avoid performing a full calibration after resetting the sampler.

- 9.5.1.4. Verification techniques are included with the following calibration procedures. When performing routine calibrations at the six-month intervals, follow all verification steps concerning ambient temperature, filter temperature, ambient pressure and flow.
- 9.5.2. Summary of the calibration procedures
 - 9.5.2.1. The control of a volumetric flow, and the monitoring of the temperature of the exposed filter while still in the sampling position is required for this sampler.
 - 9.5.2.2. To perform these tasks, the sampler uses an ambient temperature sensor (AmbT), a filter temperature sensor (FltT), a pressure sensor (Pres) and a mass flow controller (MFC). These sensors and MFC require calibration when the sampler is installed/replaced and require verification/calibration every 6 mos, or sooner if so specified in the QAPP.
 - 9.5.2.3. If the AmbT sensor and/or the Pres sensor require calibration or adjustment, then the flow must also be verified and probably recalibrated.
- 9.5.3. Apparatus for calibrating TEC PM_{2.5} FRM Single-Channel Sampler
 - 9.5.3.1. Certified flow rate standard (FRS).
 - 9.5.3.2. Certified reference temperature meter
 - 9.5.3.3. Certified pressure meter
 - 9.5.3.4. TEC inlet flow audit adapter
 - 9.5.3.5. Tubing (Tygon or surgical type)
 - 9.5.3.6. Blank filter (Teflon 47-mm diameter)
 - 9.5.3.7. Calibration forms and/or laptop computer
 - 9.5.3.8. Digital volt meter (DVM)
 - 9.5.3.9. TEC FTS Streamline 0-20 L/min fixed orifice calibration kit (Optional)
- 9.5.4. Calibration procedures outline (Note: Routine calibration will skip Sections 9.5.4.6 & 9.5.4.7)
 - 9.5.4.1. Verify/calibrate AmbT ($\pm 2^{\circ}\text{C}$).
 - 9.5.4.2. Verify/calibrate FltT ($\pm 2^{\circ}\text{C}$).
 - 9.5.4.3. Verify/calibrate Pres ($\pm 10\text{mm Hg}$).
 - 9.5.4.4. Perform leak check.
 - 9.5.4.5. Verify flow (single point verification $\pm 4\%$ of flow rate standard)
 - 9.5.4.6. Verify/calibrate interface board.
 - 9.5.4.7. Verify/calibrate analog input (A/I).
 - 9.5.4.8. Calibrate flow.
 - 9.5.4.9. Verify calibrated flow ($\pm 2\%$ of 16.7 L/min; 16.4 - 17.0 LPM).
- 9.5.5. Calibration procedures
 - 9.5.5.1. Perform an external leak check (TEC manual, Section 10.2.4) on the sampler. The leak check procedure is performed to determine if leaks in the sampler exist, and to ensure correct installation of the flow measuring equipment. If the following leak check fails, locate and remedy the problem, then re-verify the flow.
 - 9.5.5.1.1. Carefully remove and replace the 1st stage inlet impactor with the supplied flow audit adaptor and the stopcock valve open.

- 9.5.5.1.2. Start in the main screen by pressing <ESC> if necessary and make sure that the sampler is in the “stop” operating mode by pressing <F4: Run/Stp> until “stop” is displayed in the upper right hand corner.
- 9.5.5.1.3. Install a filter cassette containing a 47-mm filter into filter-holding mechanism.
- 9.5.5.1.4. Press <F5: Setup>, then <F5: Audit>, then <F2: Valve> and then <F3: Pump>.
- 9.5.5.1.5. Close the stopcock valve on the audit flow adaptor and read the vacuum gauge immediately (the vacuum gauge is located behind the small hinged door below the opening of the filter-holding mechanism).
- 9.5.5.1.6. Within 30 s, the vacuum gauge should hold at or above 8.5 in of Hg. If the vacuum gauge indicates a value less than 8.5 in of Hg during this 30-s period, locate the leak, fix and re-check.
- 9.5.5.1.7. When finished, press <ESC> twice to return to the main screen.
- 9.5.5.2. An as-is flow check verification is the first step for routine calibration of the TEC 2000 (initial sampler setup does not require an as-is flow verification since a full instrument calibration is required). The as-is flow check procedure (TEC manual, Section 10.2.5) verifies the working status of the sampler by ensuring that the calculated true flow at the inlet is $\pm 4\%$ of 16.7 L/min. If the calculated true flow of the sampler is between 16.032 and 17.4 L/min, flow calibration is not necessary unless subsequent sensor or electronics adjustments alter the flow. There are two methods to verify and calibrate flow. The first measures flow directly with a NIST-traceable flow rate standard (FRS); the second method uses the Streamline® Flow Transfer Standard (FTS), which measures a pressure differential. Both FRS and FTS data must be corrected to volumetric flow rate using temperature and pressure data
 - 9.5.5.2.1. Record all calibration measurements and details of standard/audit barometer, thermometer, and FRS calibrations on the Calibration Datasheet, Appendix E.
- 9.5.5.3. **As-is flow check using a primary flow meter** (such as the Gilian Gilibrator-2 air flow calibration system, Sensidyne Inc., Clearwater, FL) (SOP A9) (Fig. 5): This is the method of choice for verifying inlet flow because this flow rate standard measures flow directly. The following procedure describes how to verify flow with a certified FRS.
 - 9.5.5.3.1. Carefully remove and replace the 1st stage inlet impactor with the flow audit adaptor and open the stopcock valve.
 - 9.5.5.3.2. Connect the FRS flow inlet adaptor to the (Fig. 10.2 of the Sampler Operation Manual).
 - 9.5.5.3.3. Start in the main screen by pressing <ESC> if necessary and make sure that the sampler is in the “stop” operating mode by pressing <F4:Run/Stp> until “stop” is displayed in the upper right hand corner.
 - 9.5.5.3.4. Install a cassette containing a 47-mm filter into the filter-holding mechanism.
 - 9.5.5.3.5. Press <F5: Setup>, <F5: Audit>, <F2: Valve>, and then <F3: Pump>.
 - 9.5.5.3.6. Allow the sampler flow and PFM reading to stabilize, and record the measured flow reading.

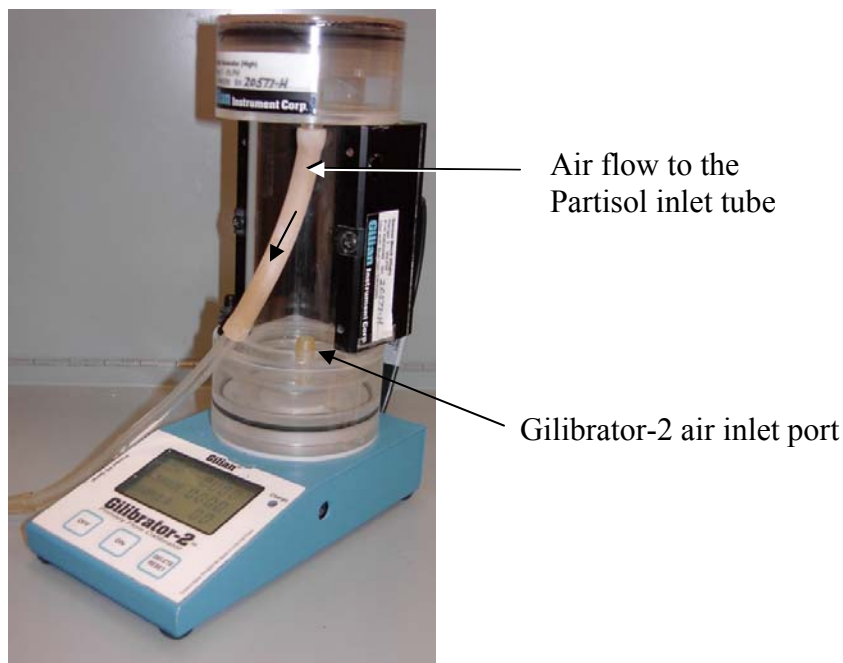


Figure 5. Gilian's air flow primary calibration system (Giliberator-2).

- 9.5.5.3.7. If a mass flow rate standard is used, calculate the true flow from the measured flow reading and determine the percent difference from 16.7 L/min. The equation used for calculating true (volumetric) flow is:

$$\text{Volumetric flow} = \frac{(\text{Std. flow}) \times T \times (760 \text{ mm Hg})}{P \times (298 \text{ }^\circ\text{K})} \quad (1)$$

Where:

T = Ambient temperature, $^\circ\text{K}$

P = Ambient pressure, mm Hg

Std. flow = [(FRS disp)(FRS cert. Slope)] + (FRS cert. Intercept)

- 9.5.5.3.8. If the calculated true flow differs more than $\pm 4\%$ of the Flow Rate Standard, the sampler flow must be recalibrated. If the flow is out of range or unstable, a leak may be the cause. Check flow check equipment to ensure it is not at fault. The calculation for % difference is:

$$\% \text{ difference} = \frac{(\text{True flow} - 16.7 \text{ L/min})}{16.7 \text{ L/min}} \times 100\% \quad (2)$$

- 9.5.5.3.9. When finished, press <ESC> twice to return to the main screen.

- 9.5.5.4. **As-is flow check using FTS:** This is the secondary method of choice when used for measuring inlet flow because the FTS measures a pressure differential and

must be converted to flow units. The FTS is an accurate device and appears to work well when used as a calibration tool, but when possible, the primary choice for verifying flow should be a flow measuring device. The following procedure describes how to use the FTS as a flow check device.

- 9.5.5.4.1. Carefully remove and replace the 1st stage inlet impactor with the FTS fixed orifice (the fixed orifice should be connected to the minus (-) port of the digital flow manometer by the supplied piece of black rubber tubing).
- 9.5.5.4.2. Start in the main screen by pressing <ESC> if necessary and make sure that the sampler is in the “stop” operating mode by pressing <F4: Run/Stop> until “stop” is displayed in the upper right hand corner.
- 9.5.5.4.3. Install a filter cassette containing a 47-mm filter into the filter holding mechanism.
- 9.5.5.4.4. With the digital manometer turned on and in "measuring in inches of water", adjust the silver colored knob at the top of the manometer (located between the + and - ports), until a reading of 0.00 in H₂O is reached (zeroing the manometer).
- 9.5.5.4.5. Press <F5: Setup>, <F5: Audit>, <F2: Valve>, and then <F3: Pump>.
- 9.5.5.4.6. Allow the sampler flow and FTS reading to stabilize and record the measured pressure reading.
- 9.5.5.4.7. Calculate true flow from the measured FTS reading and determine the percent difference from 16.7 L/min. The true flow conversion equation, orifice slope and intercept used for calculating true flow is located on the fixed orifice and is as follows:

$$\text{True flow} = \frac{(\text{orifice } m) \times (\text{FTS } P \times T)^{0.5}}{\text{AmbP}} + (\text{orifice } b) \quad (3)$$

Where:

 - orifice m = Orifice slope.
 - orifice b = Orifice intercept.
 - FTS P = FTS differential pressure reading, inches of H₂O
 - T = Ambient temperature, °K
 - AmbP = Ambient pressure, atm
- 9.5.5.4.8. If the calculated true flow differs more than ± 2% of 16.7 L/min, the TEC 2000 sampler flow must be recalibrated. If the flow is out of range or unstable, a leak may be the cause. Verify that flow check is not faulty.
- 9.5.5.4.9. When finished, press <ESC> twice to return to the main screen.
- 9.5.5.5. Interface Board Verification and Calibration (TEC manual, Section 11.1). The following procedure describes how to check and adjust the Interface (I) board.
 - 9.5.5.5.1. Sampler must be turned on and in the “stop” operation mode.
 - 9.5.5.5.2. Locate the interface board. The interface board is located on the bottom of the electronics compartment. Access the compartment by removing the three

screws on the hinged door located above the front digital display. The layout of the interface board is shown in Fig. 6.

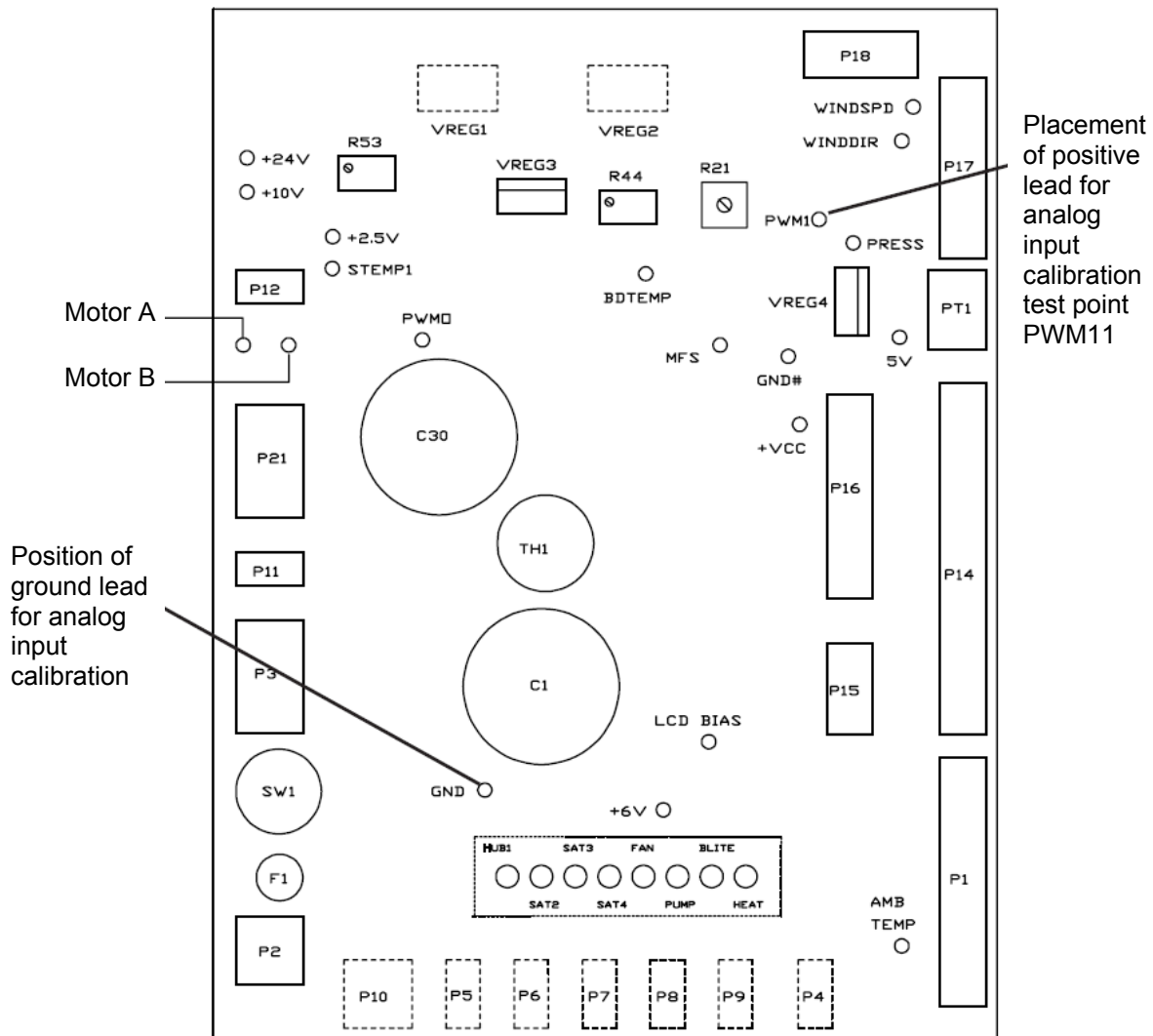


Figure 6. Layout of the interface board indicating the lead locations.

- 9.5.5.5.3. The digital display backlight must be on (pressing any key on the keypad, such as the <ESC> key, will turn on the backlight).
- 9.5.5.5.4. Locate the red 2.5-V, 6-V, 10-V and black GND test points and the adjustable pots labeled R21, R44 and R53 on the I board (refer to Fig. 6 for locations).
- 9.5.5.5.5. Place the positive lead of the DVM on the 6-V test point and the negative lead on the GND test point. Read and record the output.
- 9.5.5.5.6. If needed, adjust pot R21 until DVM reads 6.0 VDC \pm 0.05 V.
- 9.5.5.5.7. Next, place the DVM positive lead on the 2.5-V test point, read, record and adjust pot R53 until the DVM reads 2.5 VDC \pm 0.002 V.

- 9.5.5.5.8. Now, place the DVM positive lead on the 10-V test point, read, record and adjust pot R44 until the DVM reads $10 \text{ VDC} \pm 0.002 \text{ V}$.
- 9.5.5.5.9. Leave the electronics compartment door open to calibrate the analog input (A/I) board in the following Section (9.5.5.6).
- 9.5.5.5.10. The interface board calibration is now complete and should only need checking and calibration when the sampler is moved or when troubleshooting.
- 9.5.5.6. A/I verification/calibration
 - After checking/calibrating the interface board, the A/I board must be checked and calibrated as needed (TEC manual, Section 11.2).
 - 9.5.5.6.1. Push the <ESC> key on the keypad to return to main screen and ensure that the sampler is in the “stop” operating mode.
 - 9.5.5.6.2. Locate and unplug the connector cable labeled P18 at the back of the I board by gently pulling the thin brown plastic strap on top of the plug forward (refer to Fig. 6 for location).
 - 9.5.5.6.3. Plug in the analog adaptor plug into P18 (adaptor plug is provided with the sampler, TEC p/n 51-004282).
 - 9.5.5.6.4. Place the DVM positive lead on the green test point labeled PWMI, and the DVM negative lead on the test point labeled GND.
 - 9.5.5.6.5. Press <F5: Setup>, then <F2: Calib> and then <F1: Edit>.
 - 9.5.5.6.6. Move the cursor over to the right hand side of the screen to the row labeled “A/O”.
 - 9.5.5.6.7. Choose and enter a value between 0.050 and 0.150 VDC (i.e. 0.100 VDC).
 - 9.5.5.6.8. Observe the number displayed in the column labeled “calc” on the row labeled “A/I”. This number should not vary more than $\pm 0.005 \text{ V}$ (from itself) after watching for 5 s. If this number is not stable, choose and enter another value between 0.050 and 0.150 V until the number displayed becomes stable.
 - 9.5.5.6.9. With the DVM in place, read the DVM display.
 - 9.5.5.6.10. Press <F1: EDIT> and using the arrow keys, move the cursor to the column labeled “Act” in the row labeled A/I.
 - 9.5.5.6.11. Enter the DVM reading to three decimals (a.bcd) and press <ENTER> (this calculates offset).
 - 9.5.5.6.12. The number in the “calc” column should match the number on the DVM $\pm 0.005 \text{ V}$.
 - 9.5.5.6.13. Press <F1: EDIT> again and move the cursor back over to the right hand side of the screen to the row labeled “A/O”.
 - 9.5.5.6.14. Choose and enter in a number between 4.800 and 4.900 VDC (i.e. 4.850 V) and press <ENTER>.
 - 9.5.5.6.15. Observe again the number displayed in the column labeled “calc” in the row labeled “A/I”. This should not vary more than $\pm 0.005 \text{ V}$ (from itself) after watching it for 5 s (if this number is unstable, choose another number for “A/O” between 4.800 and 4.900 V until stable).
 - 9.5.5.6.16. Press <F1: EDIT> and using the arrow keys, move the cursor to the column labeled “Act” in the row labeled “A/I”.

- 9.5.5.6.17. Type in the DVM reading to three decimals and press <ENTER> (this calculates span). The number now displayed on the DVM should match the number in the column labeled “calc” within ± 0.005 V.
- 9.5.5.6.18. Remove the adaptor plug and reconnect the cable in location P18.
- 9.5.5.6.19. The A/I calibration is now complete and should only need checking and calibration when sampler is moved, when troubleshooting or when the interface board is calibrated.
- 9.5.5.7. Temperature calibration: The TEC single channel PM_{2.5} FRM sampler has two temperature sensors: the ambient and the filter sensors. These two temperature sensors require one temperature data point each to calibrate. The following procedure requires an external certified thermometer.
 - 9.5.5.7.1. Ambient Temperature Check/Calibration
 - The following procedures are found in the TEC manual, section 11.3.
 - 9.5.5.7.1.1. As-Is Ambient Temperature Check
 - 9.5.5.7.1.1.1. Remove the ambient temperature thermocouple from the external housing.
 - 9.5.5.7.1.1.2. Place the thermocouple and the external thermometer in a similar medium if possible (i.e. packing material). The main point is to place them in as similar an environment as possible.
 - 9.5.5.7.1.1.3. Record and compare sensor and external thermometer readings. The sensor reading must be within 2°C of the external thermometer temperature.
 - 9.5.5.7.1.1.4. If the ambient temperature sensor fails the ambient temperature check, calibrate the sensor.
 - 9.5.5.7.1.2. Ambient temperature calibration
 - 9.5.5.7.1.2.1. To calibrate the ambient temperature sensor, start in the main screen and ensure that sampler is in the “stop” operation mode.
 - 9.5.5.7.1.2.2. Press <F5: Setup>, then <F2: Calib> and then <F1: Edit>.
 - 9.5.5.7.1.2.3. Using the arrow keys, move cursor to the “Act” column in the row labeled “AmbT”.
 - 9.5.5.7.1.2.4. Enter the current temperature as displayed on the external thermometer device (press <SHIFT><F1> to enter a negative number).
 - 9.5.5.7.1.2.5. Press <ENTER> and record the offset number listed under the column labeled “offset” on the Datasheet, Appendix E.
 - 9.5.5.7.2. Filter temperature calibration
 - The following procedures are found in the TEC manual, Section 11.4.
 - 9.5.5.7.2.1. Filter temperature check
 - 9.5.5.7.2.1.1. The filter sensor is located directly beneath the filter cassette. Open the filter exchange mechanism by pulling on the foam padded handle and remove the filter cassette if present.
 - 9.5.5.7.2.1.2. Place the external thermometer sensor next to the filter sensor so that they share the same environment.

- 9.5.5.7.2.1.3. Record and compare sensor and external thermometer readings. The sensor reading must be within $\pm 2^{\circ}\text{C}$ of the external thermometer temperature.
- 9.5.5.7.2.1.4. If the filter temperature sensor fails the ambient temperature check, calibrate the sensor.
- 9.5.5.7.2.2. Filter temperature calibration
 - 9.5.5.7.2.2.1. To calibrate the filter temperature sensor, start in the main screen and ensure that sampler is in the “stop” operation mode.
 - 9.5.5.7.2.2.2. Press <F5: Setup>, then <F2: Calib> and then <F1: Edit>.
 - 9.5.5.7.2.2.3. Using the arrow keys, move cursor to the “Act” column in the row labeled “FltT”.
 - 9.5.5.7.2.2.4. Enter the current temperature as displayed on the external thermometer device (press <SHIFT><F1> to enter a negative number).
 - 9.5.5.7.2.2.5. Press <ENTER> and record the offset number located in the column labeled “offset” on the Calibration Datasheet, Appendix E.
- 9.5.5.8. Ambient pressure calibration
 - 9.5.5.8.1. The ambient pressure calibration (TEC manual, Section 11.5) requires a certified pressure sensor.
 - 9.5.5.8.2. Return the sampler to the Main Screen and make sure that the sampler is in the “stop” operation mode.
 - 9.5.5.8.3. Compare the displayed TEC 2000 pressure reading to the pressure of the certified pressure sensor. The sampler pressure reading should be within 10 mm Hg of the pressure measuring device. If not, then proceed to calibrate.
 - 9.5.5.8.4. Press <F5: Setup>, then <F2: Calib> and then <F1: Edit>.
 - 9.5.5.8.5. Determine the current ambient pressure in mm Hg.
 - 9.5.5.8.6. Using the arrow keys, move the cursor to “Act” column in the row labeled “Pres”.
 - 9.5.5.8.7. Enter the current ambient pressure in mm Hg and press <ENTER>.
 - 9.5.5.8.8. Record the offset number located in the column labeled “offset” on the Calibration Datasheet (Appendix E).
- 9.5.5.9. Multipoint flow calibration (TEC manual, Section 11.6.2) a minimum of three points are required. The TEC 2000 flow calibration is software driven and offers the option of a one-point or a five-point calibration. Due to U.S. EPA requirements, perform the five-point flow calibration procedure. The mass flow controller of the TEC 2000 can be calibrated using a flow rate standard (FRS) or the Streamline FTS. When using the a mass flow meter (MFM) as the FRS, flow input data must be converted from standard flow to true flow using temperature and pressure data. When using the Streamline FTS, there are no external data conversions. Both procedures are described below.
 - 9.5.5.9.1. Flow calibration using MFM:
 - 9.5.5.9.1.1. Return to the Main Screen and make sure that the sampler is in the “stop” operating mode (by press <F4: Run/Stp> if needed).
 - 9.5.5.9.1.2. Carefully remove the 1st stage inlet from sampler.

- 9.5.5.9.1.3. Carefully install the flow audit adaptor and turn the stopcock valve to the open position.
- 9.5.5.9.1.4. Install a filter cassette containing a 47-mm filter into the filter holding mechanism.
- 9.5.5.9.1.5. Attach the audit flow adaptor to the MFM.
- 9.5.5.9.1.6. Press <F5: Setup>, <F2: Calib>, <F2: FlowCal>, and then <F2: Start>.
- 9.5.5.9.1.7. After a few moments, the flow pump will start. Allow the pump to run for 5 min until both the MFM and the sampler flow display produce a stable reading. At this point, enter the true flow calculated from the MFM reading into the column labeled “Flow” and press <ENTER>. The equations for true (volumetric) and standard flow are the same as those in Section 9.5.5.3.1.7.
- 9.5.5.9.1.8. The sampler will compute span and automatically change the inlet flow for the next point.
- 9.5.5.9.1.9. Repeat Step 9.5.5.8.1.7, until the fifth flow point is entered. When the last point is entered, the sampler will automatically shut off.
- 9.5.5.9.1.10. Record the offset and span value (Appendix E).
- 9.5.5.9.1.11. Return to the main menu by pressing the <ESC> key three times.
- 9.5.5.9.2. Flow calibration using FTS:
 - 9.5.5.9.2.1. This procedure is similar to the FRS procedure, except that the measured data is collected in terms of inches of water (because it uses an orifice to create a differential pressure) in lieu of L/min. Those manometer readings do not require external conversion.
 - 9.5.5.9.2.2. Return to the Main Screen and make sure that the sampler is in the “stop” operating mode (by pressing <F4: Run/Stp> if needed).
 - 9.5.5.9.2.3. Carefully remove the 1st stage inlet from sampler.
 - 9.5.5.9.2.4. Carefully connect the FTS fixed orifice to the inlet of the TEC 2000 (the fixed orifice should be connected to the minus (-) port of the digital flow manometer by the supplied piece of black rubber tubing).
 - 9.5.5.9.2.5. Install a filter cassette containing a 47-mm filter into filter holding mechanism.
 - 9.5.5.9.2.6. Enter the slope and offset values listed on the fixed orifice by pressing <F5: Setup> and then <F1: Edit>. Arrow down to the “FTS Const M” row, type in the slope of the fixed orifice and press <ENTER>.
 - 9.5.5.9.2.7. Press <F1: Edit> again, arrow down to the “FTS Const B” row, type in the intercept of the fixed orifice and press <ENTER>.
 - 9.5.5.9.2.8. Press <ESC> until back into the main screen.
 - 9.5.5.9.2.9. To start the calibration, press <F5: Setup>, then <F2: Calib>, then <F2: FlowCal>, and then <F2: Start>.
 - 9.5.5.9.2.10. After a few moments, the flow pump will start. Allow the pump to run for a few minutes until both the FTS and the sampler flow display read a stable value. At this point, enter the manometer reading in inches of water into the column labeled “FTS” and press enter.
 - 9.5.5.9.2.11. The sampler will compute span and automatically change inlet flow for the next point.

- 9.5.5.9.2.12. Allow flow to stabilize for a few minutes and enter the next manometer reading.
 - 9.5.5.9.2.13. Repeat Sections 9.5.5.8.2.8 through 9.5.5.8.2.12 until the fifth flow point is entered. When the last point is entered, the sampler will automatically shut off.
 - 9.5.5.9.2.14. Record the offset and span value.
 - 9.5.5.9.2.15. Return to the main menu by pressing the <ESC> key three times.
 - 9.5.5.9.3. Verify the calibrated flow of the sampler. This post calibration flow check is essential to verify the accuracy of the sampler flow. Follow procedures described in Section 9.6.5.3 or 9.6.5.4 (9.6.5.3 is preferred, as stated above). Record the final flow value.
- 9.6. Sampler filter handling procedures
- 9.6.1. Overview
 - 9.6.1.1. PM_{2.5} measurements is a 24-h integrated sample, and samples will be collected (typically) once every one or three days, depending of the PM concentration and availability of the site engineer.
 - 9.6.1.2. Use care in handling the samples so that the filters arrive in the lab with the same mass that was collected at the end of the sample run. Do not contaminate the filters with fugitive particulate matter, and do not lose mass from the filters by allowing particles to shake loose from the filter or heating the filters and driving off heat-sensitive particulate matter.
 - 9.6.1.3. Accurately and completely fill in the Filter Field Sample Report (Appendix D) associated with each filter.
 - 9.6.1.4. Send the filter to the lab within the appropriate time and temperature constraints.
 - 9.6.2. Field filter preparation
 - 9.6.2.1. Keep the filter cassette assembly in its protective container until installation. Never remove the filter from the cassette.
 - 9.6.2.2. In the field office or a dust-free work environment, place an unsampled filter cassette into a filter cassette holder. Do this by removing the metal cassette cover from the bottom of the cassette. Snap the cassette into the cassette holder, leaving the top cassette cover in place to protect the filter until installation into the sampler.
 - 9.6.2.3. Take the removed metal cassette cover to the sampler.
 - 9.6.2.4. Place the filter cassette holder into a filter cassette transport container.
 - 9.6.2.5. Fill in the Filter Field Sample Report (Appendix D). Include the site name, site number, operator name, cassette ID#, scheduled sampling date and sampler serial number.
 - 9.6.2.6. Take the filter, Filter Field Sample Report Appendix D), and Monthly QC Maintenance Checksheet (Appendix C) to the field sampling location.
 - 9.6.2.7. Remove the previous filter (NOTE: Always exchange filter cassettes with the sampler in the “STOP” mode.)
 - 9.6.2.8. Remove sample from the TEC sampler within 96 h (4 d) of the end of the run.
 - 9.6.2.9. Open the door of the sampler and observe the status of the previous sample run. In the upper left hand corner, “STAT” should indicate “OK”. If there was an occurrence of any status condition, its code should be displayed (reference the

- Filter Field Sample Report and/or Section 6 of the TEC Operating Manual).
- 9.6.2.10. Observe the sampler mode condition. If the sampler has completed a sample run, the condition should indicate “DONE”, unless an error condition (“ERR”) was encountered during the sampler run.
 - 9.6.2.11. Hit the <Run/Stop (F4)> touch key. The sampler should go to “STOP” mode.
 - 9.6.2.12. Hit the <Data (F3)> touchkey. Observe and record the start time/date, total elapsed time, sample volume, and flow %CV.
 - 9.6.2.13. Observe and record the minimum, average, and maximum ambient temperature, filter temperature, and pressure. Also record the average flow results. Record the local condition code.
 - 9.6.2.14. Hit the <Pwr Data (F4)> touchkey to view and record any power interruption messages.
 - 9.6.2.15. Hit the <ESC> touchkey twice to return to the Main Screen.
 - 9.6.2.16. Record the date and time of filter removal from the sampler in the Chain-of-Custody section of the Filter Field Sample Report.
 - 9.6.2.17. Pull the handle of the filter exchange mechanism out to access the sampled filter.
 - 9.6.2.18. Visually check the filter while in the cassette for tears, pinholes, or any unusual appearance, then immediately install a metal cassette cover on the top of the filter cassette.
 - 9.6.2.19. Remove the filter holder with cassette from the sampler and place it into the filter transport container. Do not allow the filters to be shaken, dropped, or touched by any foreign object.
 - 9.6.2.20. Check the sampler for any obvious problems, such as a full water jar. Describe any obvious sampler problems on the monthly maintenance checksheet, and take corrective action before the next sample run. Note any anomalies on the Filter Field Sample Report.
- 9.6.3. Complete the Monthly Maintenance Checksheet and perform all required maintenance actions. Annotate the Monthly Maintenance Checksheet accordingly.
 - 9.6.4. Loading new filter
 - 9.6.4.1. Take the unsampled filter cassette from the filter cassette transport container and remove the top metal cassette cover from the filter cassette.
 - 9.6.4.2. Place the cassette holder in position to collect a sample.
 - 9.6.4.3. Ensure the cassette holder is seated properly on the alignment pins.
 - 9.6.4.4. Push the handle of the filter exchange mechanism inward to lock the new filter cassette into place, being careful to ensure good alignment of the upper and lower cassette seals.
 - 9.6.5. Program the Sampler for the next run
 - 9.6.5.1. From the Main Screen, hit the <Filter Set (F1)> touchkey to reprogram for the new start and stop days. Hitting the <Next Day (F3)> touchkey will advance the program to the next day and default start time. Then hit the <+Day (F4)> touchkey until the correct scheduled start date is indicated. The start time/end time should not require adjustment, unless it is being changed from the default values.
 - 9.6.5.2. Use the arrow keys to cursor down to “ID1”. Hit the <Edit (F1)> touchkey, and enter the barcode number of the new sample taken from the top of the Filter

Field Sample Report. Hit <ENTER> to program that number into the sampler's memory.

- 9.6.5.3. Hit <Escape (ESC)> to return to the Main Screen.
- 9.6.5.4. Ensure that the new programmed scheduled start date/time is indicated.
- 9.6.5.5. Hit <Run/Stop (F4)> to put the sampler in the "Wait" mode. It should display "0.0 Flow Rate", "0.0 Volume", and "000.00 Sample Time".
- 9.6.6. Post-sampling procedure
 - 9.6.6.1. In the field office or a dust-free work environment, carefully remove the sampled filter from the filter holder and place a metal cassette cover on the bottom of the filter cassette.
 - 9.6.6.2. Place the sampled filter in a cold shipping cylinder.
 - 9.6.6.3. Remove the tabs and place a set of 5°C and 25°C heat sensing strips inside the shipping cylinder.
 - 9.6.6.4. Document the date and time the samples are placed in the cooler or shipped to the lab on the Filter Field Sample Report. Note any problems in the comment section of the Filter Field Sample Report, and on the Monthly QC Checksheet.
 - 9.6.6.5. Complete the information in the Chain-of- Custody section of the "Filter Field Sample Report" (Appendix D).
 - 9.6.6.6. Place the report inside a zip-lock plastic bag to avoid condensation damage and attach the bag to the exterior of the shipping cylinder with a rubber band.
 - 9.6.6.7. Place the shipping cylinder into the freezer or ship the cylinders as required.
- 9.6.7. Additional filter handling and shipping information
 - 9.6.7.1. Some of the major differences between collection of PM_{2.5} using the new FRM and the older dichotomous filter sampling for particulate matter are the additional time and temperature requirements (see *Filter Holding Times* presented in Appendix A).
 - 9.6.7.1.1. The new FRM PM_{2.5} filters must be used for sampling within 30 d of the date of preweighing by the lab.
 - 9.6.7.1.2. Postweighing by the laboratory must be performed within 10 d of the end of the sampling if the sampled filters have been continuously stored at no more than 25°C since removal from the sampler.
 - 9.6.7.1.3. If the sampled filters have been continuously stored at 4°C or less since removal from the sampler, the laboratory can conduct postweighing within 30 d of the end of sampling.
 - 9.6.7.2. Since this latter (30 d at <4°C) requirement is preferred to give sufficient time for field, transport, and laboratory operations, additional equipment such as a freezer, insulated shipping containers, and chilled medium ("Blue Ice" or equivalent), is required.
 - 9.6.7.2.1. Store the shipping cylinders (containing the unused, ready-to-sample filters, and the used filters) in the freezer, in such a way that the first received is the first sampled, and the first sampled is the first shipped.
 - 9.6.7.2.2. To minimize the possibility of contaminating the sample filter prior to the sampling event, load the sample filter(s) at a time as close as practical to the start of the sampling event.

- 9.6.7.2.3. Although the sample cassette(s) may remain in the sampler up to 96 h (4 d) after the end of the sampling event, remove them from the sampler as soon as practical.
- 9.6.7.3. When traveling to a site, bring a chilled shipping cylinder, and a cooler with chilled medium inside.
 - 9.6.7.3.1. The shipping cylinder will be used to chill the sampled filters to less than 4°C during the transportation period to the operator's field office or shipping location.
 - 9.6.7.3.2. If a temperature threshold is exceeded during shipment, note this information in the affected sample filter's Filter Field Sample Report.
- 9.6.7.4. Prepare the filter samples for shipment.
 - 9.6.7.4.1. Remove the shipping cylinder(s) containing the oldest (earliest sample date) sample cassettes and attached Filter Field Sample Reports from the freezer.
 - 9.6.7.4.2. Open the shipping cylinder, and confirm that each sample cassette has a matching sample report.
 - 9.6.7.4.3. Orient the indicators in such a manner that the indicator window is clearly visible upon examination.
 - 9.6.7.4.4. Fill in the appropriate portions of the Chain-of-Custody sections of each Filter Field Sample Report.
 - 9.6.7.4.5. Close the shipping cylinder(s).
- 9.6.7.5. Place the shipping cylinder with its report in an insulated shipping container.
 - 9.6.7.5.1. The type of insulated shipping container needed and the amount of chilled medium used will depend on the transit time of shipping as well as the expected ambient temperatures during shipment.
 - 9.6.7.5.2. Close the shipping container and secure the lid or opening to prevent opening during shipment.
 - 9.6.7.5.3. Address the container to the analyzing laboratory and store the container in a cool place until pickup.
 - 9.6.7.5.4. Ship samples to the lab using a suitable service or carrier. Determine beforehand how long the transit time is between pick-up and arrival at the lab. Schedule the pick-up to avoid arrival on weekend or holiday dates.
- 9.7. Troubleshooting
 - 9.7.1. If a problem is encountered as a result of the review of the sample summary data, which may affect the validity of the sample, download and store the 5-min averages.
 - 9.7.2. Review the 5-min averages for operational parameters which may exceed limits of the measurement quality objectives (Appendix A).
 - 9.7.3. Also, refer to Section 12 of the TEC Manual (2004, Rev. B): Resetting the Sampler for a probable cause and remedy.
 - 9.7.4. If the perceived problem does not affect sample validity, continue to monitor the problem or correct it.
 - 9.7.5. If the problem persists, contact the manufacturer. (Section 13.1).
- 9.8. Computer hardware and software
 - 9.8.1. Data will be stored in the data acquisition computer in the site's on-farm instrument shelter (OFIS).
 - 9.8.2. The daily summary reports of this data will be sent to the site's leader via e-mail.

- 9.8.3. Computer data can also be accessed and downloaded remotely via PCAnywhere® software.

10. Data and Records Management

- 10.1. Basic information about data acquisition and control hardware and software, raw data inspection, and management of barn data is contained in SOPs B1, B2, B3 and B5, respectively.
- 10.2. Introduction to data submittal
 - 10.2.1. Once field personnel have retrieved sampling information either manually (off the sampler's display as described in Sections 7 and 8 of the Partisol-FRM 2000 Operation Manual) or electronically, the sample run information must be forwarded to the site's Principal Investigator.
 - 10.2.2. If the sampling information was recorded manually, a Filter Field Sample Report(s) will accompany the sampled filter(s) to the laboratory.
 - 10.2.3. If the sampling information was recorded electronically, the sampling information will be sent to the lab via file transfer protocol and an abbreviated Filter sample record, and additional sampling information will accompany the sampled filter(s) on the Filter Field Sample Report(s).
- 10.3. When QC checks are performed, the date, results, and any pertinent comments should be recorded onto the Monthly Quality Control Maintenance Checksheet for the FRM PM_{2.5} Filter Sampler (QC Checksheet) presented in Appendix C. It is recommended that a copy be made by the operator and kept at the field site for later reference by the operator or a site visitor.

11. Quality Control and Quality Assurance

- 11.1. Schedules for calibration, maintenance, and other QA/QC-related activities for field-deployed equipment are included in Appendix B.
- 11.2. Laboratory Quality Control and Quality Assurance Checks are listed in Appendix F.
- 11.3. Trip blanks and field blanks are included in Section 9.4.4.8.
- 11.4. The field and laboratory procedures training will be evaluated using forms included in Appendix G and Appendix H.

12. References

- 12.1. USEPA. 1997. Reference Method for the Determination of Fine Particulate Matter as PM_{2.5} in the Atmosphere. U.S. Environmental Protection Agency. 40 CFR Part 50, Appendix L.
- 12.2. USEPA. 1998. Quality Assurance Guidance Document 2.12. Available at <http://www.epa.gov/tn/amtic/files/ambient/pm25/qa/m212covd.pdf> . Accessed on January 23, 2006.

- 12.3. USEPA. 2001. Guidance for Preparing Standard Operating Procedures (SOPs). US Environmental Protection Agency. Publication QA.G-6, EPA Quality Assurance Division, Washington, DC. November. Document EPA/240/B-01/004. Available at <http://www.epa.gov/quality/qs-docs/g6-final.pdf>. Accessed on September 2006.
- 12.4. Partisol –FRM Manual. 2004. Partisol-FRM Model 2000 PM-2.5 Air Sampler Operating Manual, Rev. B.” (TEC Part No. 42-004298).
- 12.5. State of California, Air Resources Board, Monitoring and Laboratory Division. 1999. “Air Monitoring Quality assurance Volume II, Standard Operating Procedures for Air Quality Monitoring, Appendix AJ, TEC Partisol-FRM MODEL 2000 PM-2.5 AIR SAMPLER”. Online at <http://www.arb.ca.gov/aqdas/aqsbdocs1/v2apxaj.pdf>. Accessed December 21, 2005.
- 12.6. USEPA. 1998. Quality Assurance Guidance Document: Model Quality Assurance Project Plan for the PM_{2.5} Ambient Air Monitoring Program at state and Local Air Monitoring Stations (SLAMS), Document EPA-454/R-98-005, April, 1998.
- 12.7. USEPA. 1998. Quality Assurance Guidance Document; Method Compendium; Field Standard Operating Procedures for the PM_{2.5} Performance evaluation Program. Available at <http://www.epa.gov/ttn/amtic/pmqa.html> accessed on 8/22/06.
- 12.8. SOP A9. 2006. Measurement of Airflow Rate or Calibration of Air Sampling Instruments with Gilibrator 2 Calibration Systems. Standard Operating Procedure A9. Purdue Ag Air Quality Lab.

13. Contact Information

13.1. Suppliers Contact Information

Partisol instrument: Thermo electron Corporation, TEC:

Senior Customer Support Lead, Air Quality Instruments (TEC Products)

Environmental Instruments Division, Thermo Electron Corporation

Phone: 518-452-0065 Ext. 2258, Fax: 518-452-0067

<http://www.thermo.com/com/cda/product/detail/0,1055,10122676,00.html>

Appendix A. Measurement Quality Objectives for FRM PM_{2.5}

Requirement	Frequency	Acceptance Criteria
Filter Holding Times: Pre-sampling Post-sampling (in sampler) Post-sampling (during storage and transport)	All sample filters	<30 d after preweighing. Store in the freezer. <96 h from end of sampling <10 d at 25°C from end of sampling or <30 d at 4°C from end of sampling
Sampling Period:	All data	1380 to 4380 min (one to three days)
Sampler: Flow rate Flow rate variability Filter temp sensor	Every 24 hrs (or every sampling period) of operation 24 hrs 24 hrs 24 hrs	Average $\pm 5\%$ of 16.67 L/min $\leq 2\%$ CV 5-min averages variation over 24 hr $\leq (5\%$ of 16.67 L/min = 0.834 L/min) No excursions of $>5^\circ\text{C}$ lasting longer than 30 min
Data Completeness	Quarterly	75%
Filter:	All filters	Visual defect check
Monthly QC Check: Flow rate Leak check Ambient temp sensor Ambient press sensor Clock/timer	Monthly	$\pm 4\%$ of audit standard (One point verification) <5 in of Hg pd in 1 min 80 mL/min $\pm 2^\circ\text{C}$ of standard ± 10 mm of Hg of standard ± 10 min of corrected clock time
Multipoint Calibration: Flow rate Leak check Temperature Sensor Pressure Sensor	Semiannually or when failed monthly check following major repair, or after sampler transport	$\pm 2\%$ of transfer (xfer) standard <5 in of Hg pd in 1 minute $\pm 2^\circ\text{C}$ of xfer standard ± 10 min of corrected clock time
Monthly QC Standards: Flow rate standards Temperature Standard Pressure standard	Annually	$\pm 2\%$ of full scale @ 20 L/min $\pm 0.1^\circ\text{C}$ resolution $\pm 0.5^\circ\text{C}$ accuracy ± 1 mm Hg resolution ± 5 mm Hg accuracy
Calibration Xfer Standards: Flow rate xfer standard Temperature xfer standard Pressure xfer standard	FTS meter annually Mass flow meter quarterly Annually Annually	$\pm 2\%$ of NIST-traceable standard $\pm 0.1^\circ\text{C}$ resolution $\pm 0.5^\circ\text{C}$ accuracy ± 1 mm Hg resolution ± 5 mm Hg accuracy

Appendix B. Quality Control Maintenance and Calibration Schedule for Federal Reference Method PM_{2.5} Sampling Using the TEC Partisol-FRM Model 2000

	*Daily	Every 5 Samples	Every Month	Every 14 Samples	Every 6 Months	Every 12 Months
Record and review run data summary	X					
Clean/inspect cassette & rubber seals	X					
Inspect or drain inlet water jar	X					
Service WINS impactor well		X				
Perform a leak test		X	X			
Clean interior of sampler			X			
Clean air intake filter and fan			X			
Inspect O-rings, gaskets and seals			X			
Verify sampler clock time			X			
Perform single-point flow check			X			
Check ambient temperature and pressure sensors			X			
Run field and trip blanks			X			
Transport samples with a temp-logger			X			
Disassemble and clean PM ₁₀ inlet, downtube and entire WINS				X		
Perform as-is three-point calibration/verification of flow rate, pressure and temperature sensors					X	
Verify as-is condition of sampler interior, inlet, WINS impactor & leak test					X	
Replace large in-line filter					X	
Check battery on main computer board					X	
Perform multi-point calibration of flow rate, pressure & temperature sensors					X	
Measure temp of station freezer	X					
Calibrate or re-certify flow rate, pressure and temperature QC check standards						X

*or each time sample cassettes are exchanged

Appendix C. Monthly Quality Control Maintenance Check Sheet TEC Partisol – FRM Model 2000 PM_{2.5} Air Sampler Single Channel

Site Name _____ MM/DD/YYYY _____
 Site Number _____ Technician _____
 Sampler I.D. Number _____

Operator Instructions:

Each Run: Record and review sample run date; inspect/clean water jar, cassette & rubber seals.

Run Dates: _____

Every 5 Runs: Clean or change out WINS impactor well.

Dates WINS Serviced: _____

Monthly: Clean interior of sampler, air intake filter and fan. Date Performed: _____

Inspect o-rings, gaskets, seals; check sampler clock time ($\leq \pm 10\text{ min}$): _____

Perform single-point QC check of flow rate, ambient temperature and pressure. * (annotate below)

Run field and trip blanks, and temp-logger with samples during transport. Date: _____

Perform leak check –Date/Results: _____/_____/_____/_____/_____/_____

(<math>< 80\text{ mL/min}</math> for 1 min.: which is equal to <math>< 5''\text{ Hg}</math> change/min.)

Every 14 Runs: Disassemble and clean PM₁₀ Inlet, downtube and entire WINS. A leak test must be performed every time the WINS impactor is changed out or cleaned.

Semiannually: Perform multi-point calibration of flow rate, and single point calibration of temperature and pressure sensors.

Date Last Calibrated: _____

Perform Leak Test, Inspect sampler’s inlet, WINS, and interior conditions.

Replace large in-line filter. Date Last Performed: _____

Check Voltage Level on Main Computer Board Battery. Date Last Performed: _____

Annually: Re-certify QC check standards – Certification Date: _____

(flow std)

(temp std)

(press std)

***Monthly Sampler Flow Rate, Ambient Temp and Pressure Check Results:**

	Flow Rate Standard	Temperature Standard	Pressure Standard
Standard Name/Type:			
Identification Number:			
Correction Factor:			
Std’s Indicated Readings:			
Std’s Corrected Readings:			
Sampler’s Reading:			
Percent Difference:			
Acceptance Criteria:	15.84 to 17.50 L/min	$\leq \pm 2^\circ\text{ C}$ of standard	$\leq \pm 10\text{ mm Hg}$ of std

Operator Comments: _____

Reviewed by: _____ Date: _____

Appendix D. Filter Field Sample Report and Chain-of-Custody Form for the TEC FRM Partisol-FRM Model 2000 PM_{2.5} Sampler

Sample ID: _____ Check if data electronically submitted to Laboratory
 Site Name: _____ Cassette I. D. Number: _____
 Site Number: _____ Scheduled Sampling Date: _____
 Field Technician: _____ Sampler Property #: _____

SAMPLE SUMMARY

Start Date / Time: _____ / _____
 Total Elapsed Time: _____ Hr:min
 Volume: _____ (m³)
 Flow CV _____ (%)

Ambient Temp(C): _____
 Filter Temp (C): _____
 Pressure (mmHg): _____
 Flow (L/min): _____

MIN	AVG	MAX

Local Condition Codes: _____

Sampler Flag Codes: _____

A. No Unusual Conditions	E. Fire Nearby
B. Wind/Blown Sand/Dust	F. Sampler Malfunction
C. Construction Nearby	G. Rain
D. Farming Operation Nearby	H. Other (See Comments)

OK Good	T. Filter Temp
F. Flow Rate	I. Inst. Elec. Temp
X. Flow Cutoff	V. Power Outage
S. Ambient Temp.	E. Elapsed Sample Time
P. Ambient Pressure	C. Percent CV

Operator Comments: _____

CHAIN OF CUSTODY

ACTION	DATE MM/DD/YYYY	TIME HH:mm	FILTER TEMP. (C)	NAME
Filter shipped to site				
Filter received at site				
Sample load				
Sample removal				
Sample placed in cooler				
Sample shipped to Lab				
Sample received at Lab				
Start post-conditioning				

FOR LABORATORY USE ONLY

Postweight by:

	Mass:	Dup Mass:	Date:	Analyst:
Prewrite				
Postweight				

LabComments: _____

Appendix E. TEC PM_{2.5} FRM Partisol-FRM Single Channel Sampler Calibration Datasheet

Site Name _____ Site Number _____ Sampler Property Number _____
 Site Elevation _____ Ambient Temperature _____ Ambient Pressure _____
 Cassette I.D. _____

Temperature Sensor Calibration

Temperature Standard ID Number _____ Certification Date _____
 Ambient Temperature Sensor: Ambient Reading _____ Span _____ Offset _____
 Filter Temperature Sensor: Ambient Reading _____ Span _____ Offset _____

Pressure Sensor Calibration

Pressure Standard ID Number _____ Certification Date _____
 Standard Slope _____ Standard Intercept _____
 Barometric Pressure _____ Span _____ Offset _____

Leak Test

External Leak Flow _____ Time _____
 Internal Leak Flow _____ Time _____

Flow Calibration

MFM Standard ID Number _____ Certification Date _____
 Standard Slope _____ Standard Intercept _____

Nominal Flow	Sampler Flow	Standard Flow	Temperature
16.7	_____	_____	_____
17.5	_____	_____	_____
15.8	_____	_____	_____
18.3	_____	_____	_____
15.0	_____	_____	_____

Sampler Span _____ Offset _____
 Calibrated by _____ Checked by _____

Temperature Calibration Verification

Temperature Standard ID Number _____ Certification Date _____
 Ambient Temperature Sensor: Ambient Reading _____ Span _____ Offset _____
 Filter Temperature Sensor: Ambient Reading _____ Span _____ Offset _____

Pressure Verification

Sampler Pressure _____ Pressure Standard _____

Flow Verification

Sampler Flow _____ MFM Standard Flow: Qstd _____ Qact _____
 Percent Difference _____

Time Verification

Time Standard Make _____ Model _____ Certification Date _____ Bias _____
 Sampler Time _____ Time Standard _____ Time difference _____

Final Time Set

Sampler Time _____ Time Standard _____ Time Difference _____
 Calibrated by _____ Checked by _____

Appendix F. Lab Quality Control Maintenance Schedule for Federal Reference Method PM_{2.5} Sampling Using the TEC Partisol-FRM Model 2000

Requirement	Frequency	Acceptance Criteria	CFR, 2.12 reference†	Information provided
Blanks				
Lab blanks	Approximately 10% per weighing run	±15 µg difference	Part 50, App. L, Sec. 8.2 2.12, Sec. 7.7	Laboratory contamination
Calibration Verification				
Balance calibration	1/yr	Manufacturer's spec.	2.12, Sec. 7.2	Verification of equipment operation
Lab temp. calibration	3 mo	±2 °C	Quality assurance project plan (QAPP), Sec. 13/16	Verification of equipment operation
Lab humidity calibration	3 mo	±2%	QAPP, Sec. 13/16	Verification of equipment operation
Accuracy				
Balance audit	1/yr	±15 µg for unexposed filters	2.12, Sec. 10.2	Laboratory technician operation
Balance check	Beginning, every 10th sample, end	<3 µg	2.12, Sec. 7.14	Balance accuracy/stability
Calibration standards				
Working mass standards	3-6 mo	25 µg	2.12, Sec. 4.3 and 7.3	Standard verification
Primary mass standards	1/yr	25 µg	"	Primary standards verification
Temperature standard	1/yr	±0.1 °C resolution	2.12, Sec. 4.3.7 and 7.6	Standards verification
		±0.5 °C accuracy		(check of accuracy)
Relative Humidity std	1/yr	±2% RH accuracy,	2.12, Sec.4.3.7 and 7.6	Standard verification
		±0.5%RH resolution		
Precision				
Replicate filter weighing	1 per weighing session	±15 µg difference	2.12,	Weighing repeatability/filter stability
Primary flow meter calibration	1/yr	±2% of NIST-traceable Std.	Part 50, App. L, Sec.9.1 & 9.2	Standard verification

† 2.12 refers to the EPA's Quality Assurance Guidance Document 2.12, Monitoring PM_{2.5} in Ambient Air Using Designated Reference or Class I Equivalent Methods. November 1998.

Appendix G. Training Certification Evaluation Form. Field Sampling Procedures.***Training Certification Evaluation Form***
Field Sampling Procedures

Trainee: _____

Date _____

Evaluator: _____

Score: _____

Activity	Successful	Comment
Prepare for Site Visit on Scheduled Date/time		
1) Preweighed sampling filter in cassette, packed in a labeled carrier. Also take spares.		
2) Three preweighed field blank filters in cassettes, packed in labeled carriers, if a field blank study is scheduled		
3) PM _{2.5} Sampler Run Data Sheet for each sampler, site notebook; calculator		
4) Transfer standard for ambient temperature measurements		
5) Transfer standard for ambient atmospheric pressure measurements		
6) Transfer standard for volumetric flow-rate measurements		
7) Laptop computer and connecting cables to download sampler data		
8) Spare parts and tools to include O-rings, silicone grease, lab wipes, voltmeter, etc.		
9) Operator's manual for sampler(s) to be serviced		
SCORE	/9	
Fifth Day Maintenance Check		
1) Clean impactor well assembly or filter/lab wipes/diffusion oil to clean and service the one at the site		
2) Sample inlet adapter and flow rate measurement transfer standard		
3) Clean, unused flow check filter in its cassette		
4) Sampler Flow Check Data Sheet		
SCORE	/4	
Install Filter/Cassette and Begin Sampler Operations		
1) Remove the new filter/cassette from its protective case and visually inspect the filter/cassette for flaws. Verify that this is the correct filter for this sampler, site, and run date		
2) Be sure sampler is not operating.		
3) Fill in initial information on PM _{2.5} Run Data Sheet.		

Appendix G Training Certification Evaluation Form. Field Sampling Procedures. (Cont.)

Activity	Successful	Comment
4) Remove the sampler's filter holder assembly (if required by the manufacturer's instructions). Inspect the O-rings inside the filter holder.		
5) Install the filter/cassette in the filter holder assembly, and then install the loaded filter holder assembly in the sampler per the manufacturer's instructions. If you touch or scratch the filter, void the filter and get another one from the set of extra filters brought to the site.		
6) Program the sampler to energize at the beginning of a sampling period (consult the instruction manual).		
7) Make independent measurements of ambient temperature (T_a) and ambient pressure (P_a) using transfer standards. Record these values and the T_a and P_a values indicated by the sampler on the data sheet		
8) Ensure that the sampler(s) begins operation at the designated time. Record the start time on the data sheet. 15 minutes after sampling begins, record the sampler's display value for the indicated flow rate, Q , in L/min on the data sheet.		
SCORE	/8	
Remove Filter/Cassette; End Sampling Operations		
1) Determine P_a and T_a using transfer standards. Enter on data sheet.		
2) When sampling ends, record stop time, total elapsed time, final Q , Q_{avg} , Q_{cv} , total volume sampled, T_a , P_a , etc. on data sheet		
3) After each completed run, download data from the sampler data port to a laptop or other computer storage disk.		
4) Open the filter holder assembly (consult the instruction manual); remove the used filter/cassette; visually inspect the filter for tears, oil, insects, moisture, etc; and record observations on the data sheet.		
5) Place the filter/cassette inside a properly labeled protective container. Verify the container's label versus the site name, date, etc.		
6) Place the container inside a cooled storage chest. Do not allow the metal container to come into contact with ice or water. Sealed cooling blocks are recommended. Protect the containers from condensed water.		
7) Inspect the interior of the filter housing. Note any abnormalities.		
8) Inspect the interior of the impactor housing and the exterior of the impactor well. Remove any moisture or dust with a lint-free wipe and make notes on the data sheet.		
9) Without opening the impactor well, inspect the well's interior. Note any abnormalities. Clean or replace the impactor well if necessary or if the recommended 5-day servicing is due. Reinstall the impactor assembly. (If another sampling run is to begin, insert a new filter/cassette in the filter holder assembly and set up the sampler for the next run.)		

Appendix G Training Certification Evaluation Form. Field Sampling Procedures. (Cont.)

Activity	Successful	Comment
10) Review the recorded data for sample elapsed time, flow rate, filter quality, and temperature to start the process of determining if the sample is valid, questionable, or invalid. Scan through the sampling summary on the sampler display and note flags. Record observations and reasoning for questioning or invalidating a run on the data sheet.		
11) Make a final check of the site, and observe and record the presence of any activity that may have affected the particulate loading of the sample.		
12) Keep the container holding the filter/cassette at a temperature of less than 25 °C (preferably cooled to 4 °C), and promptly deliver it and the original of the data sheet to the sample custodian in receiving facility. Keep a copy of the data sheet with the site records.		
SCORE	/12	
FINAL SCORE	/33	
PERCENTAGE	%	

Training Certification Evaluation Form source: Quality Assurance Guidance Document: Model Quality Assurance Project Plan for the PM_{2.5} Ambient Air Monitoring Program at state and Local Air Monitoring Stations (SLAMS), Document EPA-454/R-98-005, April, 1998.

Appendix H Training Certification Evaluation Form. Laboratory Procedures.***Training Certification Evaluation Form***
Laboratory Procedures

Trainee: _____

Date _____

Evaluator: _____

Score: _____

Activity	Successful	Comments
Pre-sampling PROCEDURES		
1) Clean the microbalance's weighing chamber with a fine brush, if necessary.		
2) Zero (i.e., tare) and calibrate the microbalance according to the manufacturer's directions. Record the tare weight on the laboratory data form and in the laboratory notebook or database.		
3) Using smooth, nonserrated, nonmetallic forceps, weigh two working mass reference standards as a QC check. Wait until the microbalance's display has remained steady for 30 to 60 seconds or until the microbalance indicates that a stable reading has been obtained. Record the certified and measured values of these standards on the laboratory data form and in the laboratory notebook or database.		
4) Record the relative humidity and temperature of the conditioning chamber on the laboratory data form and in the laboratory QC notebook or database. Verify the filter has been conditioned for at least 24 hours.		
5) Laboratory blank filters and the current sampling interval's field blank filters will be weighed at least once in each weighing session. If many filters are weighed, you may want to weigh the set of laboratory blanks more than once. A new set of three laboratory blanks will be established for each distinct filter lot		
6) Weigh the filters. Operate the balance according to the balance manufacturer's directions. Take the filter from its filter-handling container (petri dish or equivalent) by gently slipping the filter-handling forceps under the outer polyolefin support ring. Hold the filter only by the ring. Place the filter, reinforcing ring side up, next to a ²¹⁰ Po antistatic strip for 30 to 60 seconds. The antistatic strip will be inside the weighing chamber or as close to the chamber door as is practical. Immediately transfer the filter to the microbalance's pan and close the weighing chamber door. After the microbalance's display has remained steady for at least 60 seconds or until the microbalance indicates that a stable reading has been obtained, record the balance number, the sampler number the filter is intended to be used with, the filter number, the filter lot number, and the filter's tare weight (pre-sampling mass) on the laboratory data form.		

Appendix H Training Certification Evaluation Form. Laboratory Procedures (Cont.)

Activity	Successful	Comments
7) After every tenth filter weighing, the analyst will rezero the microbalance and reweigh one working standard. Record the zero and working standard measurements on the laboratory data form and the laboratory QC notebook or database. If the zero and working standard measurements disagree from the first measurements of the day by more than 3 µg (i.e., three times the microbalance's reproducibility), repeat the zeroing process and reweigh the working standards. If the two measurements still disagree, contact the laboratory's QA Officer, who may direct the analyst to (1) reweigh the previously weighed filters and/or (2) troubleshoot or repair the microbalance, re-zero and reweigh the two working standards and repeat the weighing session.		
8) Any unused filter whose weight is outside the normal range (i.e., 110 to 160 mg) must be investigated. If there is a consistent negative replication (>15 µg) for laboratory blank filters, it is usually a sign that the filters have not equilibrated long enough. In this case, notify the QA Officer.		
9) Return the filter to the filter-handling container, replace the lid, and return it to storage.		
10) Prior to filters being taken to the sites, install each filter in a filter cassette, and put the filter/cassette assembly into a protective container for transport to the sampler. Attach a label with the sampler number and the unique filter number to the outside of the protective container. This label will also be used to identify the upcoming sample run date. Record the sampler number, sample date, and filter number on the PM _{2.5} Sampler Run Data Sheet. Double-check the entries in the laboratory data form. Prepare several extra filters in case a filter is invalidated during the installation process.		
SCORE	/10	
Post-sampling DOCUMENTATION/INSPECTION PROCEDURES		
1) Examine the field data sheet. Determine whether all data needed to verify sample validity and to calculate mass concentration (e.g., average flow rate, ambient temperature and barometric pressure, and elapsed time) are provided. If data are missing or unobtainable from a field operator or if a sampler malfunction is evident, flag the filter and record in the laboratory data form that the sample has been flagged and the reason. Notify the QA Officer		

Appendix H Training Certification Evaluation Form. Laboratory Procedures (Cont.)

Activity	Successful	Comments
2) If the shipment was to be kept cold, verify that the temperature of the cooler's interior was maintained at the desired point, usually less than 4 °C. If the protective container is cold, allow it to warm to the filter conditioning environment's temperature before opening, to preclude water condensation on a cold filter. Remove the filter from its protective container and examine the container. If particulate matter or debris is found in the protective container after the filter has been removed, flag the filter and record notes on the laboratory data form that the sample has been flagged and the reason. Save the filter for inspection by the QA Officer.		
3) Match the sampler number with the correct laboratory data form on which the original microbalance number, filter number, pre-sampling filter weight, and other information were inscribed. Group filters according to the microbalance used to determine their initial tare weights. Initial separation of filters in this way will eliminate the risk of a measurement error that could result from the use of different microbalances for pre- and post-sampling weighings.		
4) Remove the filter from both the protective container and the filter cassette. Be careful not to touch or otherwise disturb the filter and its contents. Transfer the filter to a filter-handling container labeled with the corresponding filter number. Place the used filter in the container "dirty-side" up. Keep the particles from contact with the walls of the container. The filter must be handled with clean, smooth forceps and must not be touched by hands. Inspect the filter for any damage that may have occurred during sampling. If any damage is found, void the sample, and record on the laboratory data form that the sample has been voided and why. Retain the filter for inspection by the QA Officer.		
5) Transfer the filter in its filter-handling container to the conditioning chamber under the same conditions as pre-sampling ($\pm 5\%$ RH)		
6) Allow the filter to condition for not less than 24 hours		
SCORE	/6	
POST SAMPLING FILTER WEIGHING		
1) Group filters according to the microbalance used for pre-weighing and by their filter numbers. Reweigh each filter on the same microbalance on which its pre-sampling weight was obtained.		
2) Clean the microbalance's weighing chamber with a fine brush, if necessary.		
3) Zero (i.e., tare) and calibrate the microbalance according to the manufacturer's directions. Record the tare weight on the laboratory data form and in the laboratory notebook or database.		

Appendix H Training Certification Evaluation Form. Laboratory Procedures (Cont.)

Activity	Successful	Comments
4) Using smooth, nonserrated, nonmetallic forceps, weigh two working mass reference standards as a QC check. Wait until the microbalance's display has remained steady for 30 to 60 seconds or until the microbalance indicates that a stable reading has been obtained. Record the certified and measured values of these standards on the laboratory data form and in the laboratory notebook or database.		
5) Record the relative humidity and temperature of the conditioning chamber on the laboratory data form and in the laboratory QC notebook or database.		
6) Laboratory blank filters and the current sampling interval's field blank filters will be weighed at least once in each weighing session. If many filters are weighed, you may want to weigh the set of laboratory blanks more than once. A new set of three laboratory blanks will be established for each distinct filter lot		
7) Weigh the filters. Operate the balance according to the balance manufacturer's directions. Take the filter from its filter-handling container (petri dish or equivalent) by gently slipping the filter-handling forceps under the outer polyolefin support ring. Hold the filter only by the ring. Place the filter, reinforcing ring side up, on a ²¹⁰ Po antistatic strip for 30 to 60 seconds. The antistatic strip will be inside the weighing chamber or as close to the chamber door as is practical. Immediately transfer the filter to the microbalance's pan and close the weighing chamber door. After the microbalance's display has remained steady for at least 60 seconds or until the microbalance indicates that a stable reading has been obtained, record the balance number, the sampler number the filter is intended to be used with, the filter number, the filter lot number, and the filter's tare weight (pre-sampling mass) on the laboratory data form.		
8) After every tenth filter weighing, the analyst will rezero the microbalance and reweigh the one working standard. Record the zero and working standard measurements on the laboratory data form and the laboratory QC notebook or database. If the zero and working standard measurements disagree from the first measurements of the day by more than 3 µg, repeat the zeroing process and reweigh the working standards. If the two measurements still disagree, contact the laboratory's QA Officer, who may direct the analyst to (1) reweigh the previously weighed filters and/or (2) troubleshoot or repair the microbalance, re-zero and reweigh the two working standards and repeat the weighing session.		
9) Any unused filter whose weight is outside the normal range (i.e., 110 to 160 mg) must be investigated. If there is a consistent negative replication (>15 µg) for laboratory blank filters, it is usually a sign that the filters have not equilibrated long enough. In this case, notify the QA Officer.		
10) Return the filter to the filter-handling container, replace the lid, and return it to storage.		

Appendix H Training Certification Evaluation Form. Laboratory Procedures (Cont.)

Activity	Successful	Comments
11) If the pre- and post-sampling weights for the laboratory and field filter blanks disagree by more than 15 µg, repeat the measurements. If the two measurements still disagree, contact the laboratory's QA Officer, who may direct the analyst to (1) reweigh the previously weighed filters and/or (2) troubleshoot or repair the microbalance, then reweigh.		
12) If the filter will receive further analysis, return it to the filter-handling container and note on the container and the laboratory data form that additional analyses are required. Transfer the filter to the laboratory responsible for performing the additional analyses.		
134) A filter's post-sampling mass minus its pre-sampling mass is the net mass loading for that filter. Record this value on the laboratory data form. Refer to Section 11.0 of <i>Guidance Document 2.12</i> for the calculations required to compute and report ambient PM _{2.5} concentrations in µg/m ³ .		
SCORE	/13	
FINAL SCORE	/29	
PERCENTAGE	%	

Training evaluation forms source: Quality Assurance Guidance Document: Model Quality Assurance Project Plan for the PM_{2.5} Ambient Air Monitoring Program at state and Local Air Monitoring Stations (SLAMS), Document EPA-454/R-98-005, April, 1998.

**CONTINUOUS MEASUREMENT OF PM₁₀ PARTICULATE
MATTER USING THE THERMO ELECTRON SERIES
FH 62C14 (BETA) SAMPLER**

Standard Operating Procedure (SOP) P4

**CONTINUOUS MEASUREMENT OF PM₁₀ PARTICULATE MATTER
USING THE THERMO ELECTRON SERIES
FH 62C14 (BETA) SAMPLER
Standard Operating Procedure (SOP) P4**

Prepared by

Juan Carlos Ramirez

Reviewed by

Ji-Qin Ni, Bill W. Bogan, and Albert J. Heber

Effective Date: November 6, 2006

**PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)
Agricultural and Biological Engineering, Purdue University
West Lafayette, IN 47907**

Table of Contents

	Page
1. Scope and Applicability.....	5
2. Summary of Method.....	5
3. Definitions & Acronyms.....	7
4. Health and Safety.....	8
5. Cautions.....	8
6. Interferences.....	9
7. Personnel Qualifications.....	9
8. Equipment and Supplies.....	10
9. Procedure.....	10
10. Data and Records Management.....	27
11. Quality Control and Quality Assurance.....	28
12. References.....	29
13. Contact Information.....	29

Thermo C-14 Beta Sampler Calibration Form
For Samplers Using Firmware V2.049

Site: _____ Monitor S/N: _____ Date: _____

Temperature Sensor Calibration

Article I. Allow unit to run, calibrate sensors at normal system operating temperatures.
Article II. Calibration Menu – unlock code 147
Article III. Sensors submenu – go through each item in turn and adjust as needed. Log the final results after adjustment.

T₁ ambient sensor _____ Reference Ambient Temp _____ Criteria: ± 6 °C
T₂ upper head sens. _____ Reference upper head _____
T₃ lower head sens. _____ Reference lower head _____
T₄ heated tube sens. _____ Reference heated tube _____

Pressure Sensor Calibration

P₃ ambient _____ Ref. Barometer _____ Criteria: ± 10 mmHg

Note: hPa = mm.Hg * 1.33 , mm.Hg = hPa * .75218 (@20° C)

Flow Rate Calibration

Flow Device Type _____ S/N _____

Sampler Flow _____ Actual Flow _____ Criteria: ± 2%

Note: L/m = L/hr / 60

High Voltage Automatic Set Process

2. Service Menu – unlock keypad - Mechanical Control – turn pump off
3. Perform Filter Change & Zero (FC+Z)
4. Watch for QLF figure to have “**” next to it
5. Calibration Menu – unlock code 147 – High Voltage & Thresholds – Start – allow 5 minutes until stable, log readings in chart provided here
6. Back up on screens and Save to EEPROM
7. Service Menu – Mech. Ctrl – pump on, zero tape, back up & lock keypad, exit menu.

New HV

S_{ref}

Plateau Slope

Mass Foil Calibration

- 1) Operations Menu – unlock keypad if locked – Temp Ctrl System screen – set heater control to Off
- 2) Let sampler run and wait for T₂ = T₁ although being within 2°C seems to be sufficient
- 3) Service Menu – Mechanical Control screen – turn pump off.
- 4) Calibration Menu – Foil Calibration – follow screen prompts step by step
- 5) Note the Amp Values and log them in the chart to the right
- 6) Remove span foil and holder per prompt
- 7) Back up and Save to EEPROM
- 8) Operations Menu, Temp Ctrl – set heater control to On.
- 9) Service Menu, Mechanical Control – ensure that pump is On
- 10) Lock keypad

Foil Span Value μg^*	Old Amp Value	New Amp Value	Bias % of FS
---	----------------------	----------------------	---------------------

** For reference only – from the span foil in the calibration kit used for this test.*

Values Archived to EEPROM? _____

Comments:

Operator: _____

Date: _____

QA Coordinator: _____

Date: _____

Appendix B. Monthly Maintenance Checks – Thermo Electron FH 62C14 (Beta)
Continuous Ambient Particulate Monitor30

Appendix B. Monthly Maintenance Checks – Thermo Electron FH 62C14 (Beta)
Continuous Ambient Particulate Monitor31

Comments:.....31

1. Scope and Applicability

- 1.1. The Thermo Electron Corporation (TEC) Series FH62C14 (Beta) Continuous Ambient Particulate Monitor (hereafter referred as C14 Beta) will be used for continuous monitoring of the particulate matter (PM₁₀) concentrations in ambient air outside of confined animal houses or barns. Specifically, the location of the instrument is on top of the on-farm instrument shelter (OFIS).
- 1.2. The C14 Beta instrument is designated by USEPA as an equivalent method (EPA Designation No. EQPM-1102-150) for PM₁₀ (particles ≤ 10 μm in diameter).
 - 1.2.1. The instrument can be configured to measure PM_{2.5} and TSP (Total suspended particulates) as well, but this SOP does not address these applications.
- 1.3. The measurement range of the C14 Beta monitor is 0 to 5,000 μg/m³ (default) and 0 to 10,000 μg/m³, with a minimum detection limit of 1 μg/m³ (24-h average) and 9 μg/m³ (measured values). Maximum particulate load on the filter is typically 1,500 μg.
- 1.4. Use of the C14 Beta monitor is appropriate for air temperatures in the range of -20 to 40°C.

2. Summary of Method

The C14 Beta monitor (Fig. 1) works by drawing ambient air, including suspended particles, through the PM₁₀ inlet head at a flow rate that is adjusted to 16.7 L/min (1 m³/hr). The inlet head consists of a series of impaction plates, which segregate the suspended particulate matter by size. Air is deflected downwards into the acceleration jet of the impact unit. Because of their greater momentum, particles larger than the 10-μm cutoff impact out and are retained in the middle plenum impaction chamber. The particle fraction smaller than 10 μm is carried upward by the air flow, and down the vent tubes to the C14 Beta gauge sampler, where the PM₁₀ particles are deposited on a glass fiber filter tape (Fig. 2). Low-level beta radiation is emitted from the Carbon-14 radioactive source, and passes through the filter tape and deposited particles. The increase of particles collected on the tape causes a lower beta-ray measurement in the measuring chamber (Fig. 2). This filter-spot position results in a continuous observation of the increasing particulate mass, which can be converted to continuous particulate concentration data. A compensation chamber receives an equal portion of the emitted beta rays, and includes a foil with the same absorptivity as clean filter tape. This, therefore, provides a reference point for comparison with the tape that includes accumulated sample. Because of this, the instrument is less sensitive to temperature, pressure, and humidity fluctuations than some other types of continuous PM monitors. By measuring the accumulated mass of particles on the tape and the volumetric flow rate of air through the instrument, the onboard computer can calculate the PM₁₀ mass concentration in the air. Output from the C14 Beta monitor is sent to the data acquisition system located in the OFIS.

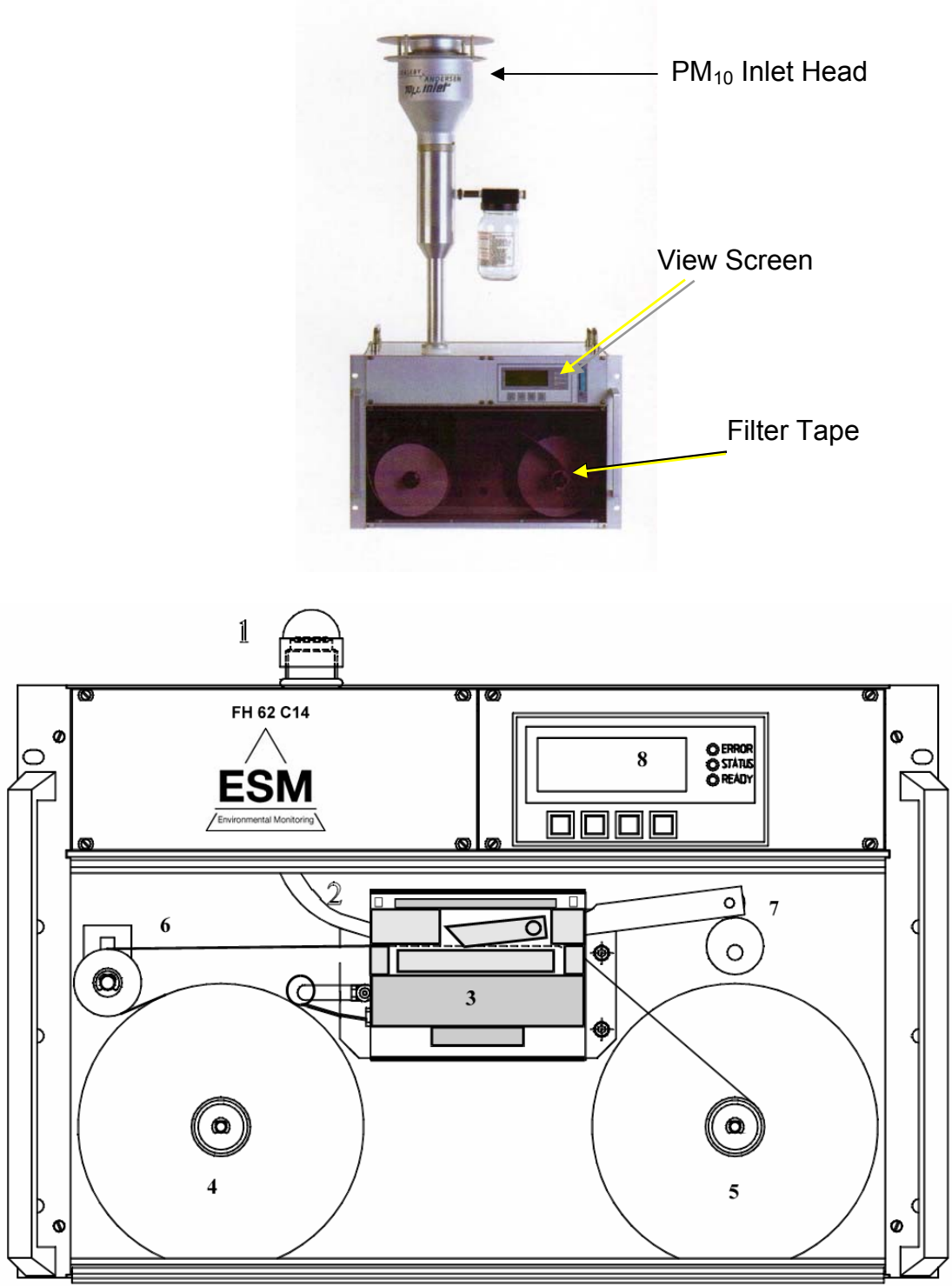


Figure 1. Thermo Electron Corporation C14 Beta Gauge (Pacwill Environmental, 2003 (photo), and Thermo Electron Corp. instrument manual (diagram)).

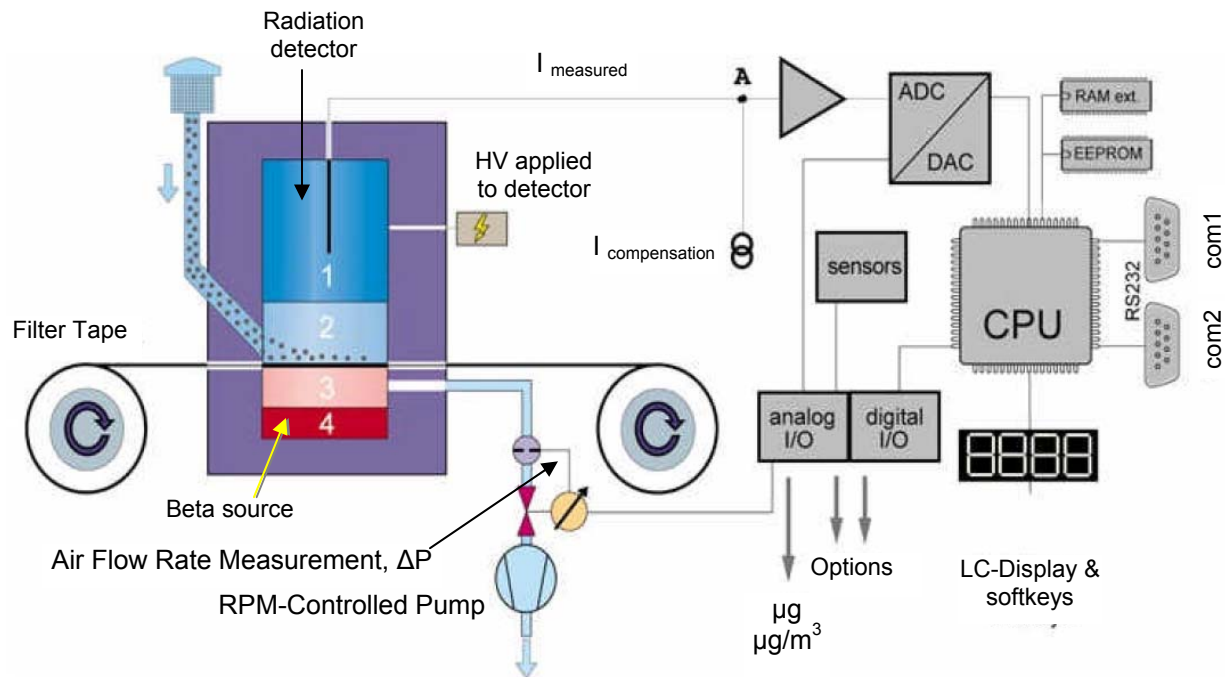


Figure 2. Thermo Electron Series FH62C14 (Beta) PM₁₀ sampler schematic (Thermo Andersen, 2002).

3. Definitions & Acronyms

- | | | |
|-------|----------------|---|
| 3.1. | AC | Alternating Current |
| 3.2. | Beta radiation | A stream of beta particles (which can be either electrons or positrons, depending on the specific emitter) emitted at close to light speed by radioactive decay |
| 3.3. | atm | atmosphere, 746 mBar |
| 3.4. | CFR | Code of Federal Regulations |
| 3.5. | C14 | Carbon-14, a low level radioactive source of beta radiation. |
| 3.6. | DAQ | Data acquisition |
| 3.7. | FTS | Flow Transfer Standard |
| 3.8. | hPa | Hecto Pascals ($\text{Pa} \cdot 10^2$) |
| 3.9. | HV | High voltage |
| 3.10. | IM | Instruction manual |
| 3.11. | I/O | Input-output |
| 3.12. | LR | Linear regression |
| 3.13. | OFIS | On-farm instrument shelter |
| 3.14. | Pa | Pascal |
| 3.15. | PAAQL | Purdue Agricultural Air Quality Laboratory |
| 3.16. | PM | Particulate matter |

3.17.	PM ₁₀	PM of ≤ 10 μm diameter
3.18.	QAPP	Quality Assurance Project Plan
3.19.	RC	Digital filter time constant for mass signal (IM Section 3.4.1)
3.20.	STDEV	Standard deviation
3.21.	SOP	Standard Operating Procedure
3.22.	TEC	Thermo Electron Corporation

4. Health and Safety

- 4.1. Instrument repair should only be carried out by trained personnel, and in accordance with procedures detailed in the IM.
- 4.2. Switch off the power supply of the instrument and unplug the power cable before opening the instrument and removing the detector.
- 4.3. Use protective eyewear when opening the detection chamber for maintenance.
- 4.4. Carbon-14 Source: Only trained personnel with radiological authorization (e.g., the TEC service division) may work on the source (e.g., cleaning the measurement chambers). The source has been secured with tamper-proof hardware and may not be disassembled without authorization.

5. Cautions

- 5.1. Do not block the sample tube or inlet of the instrument. The instrument has very sensitive internal foils. These foils and the detector window can be damaged if the air inlet is blocked when the pump is running.
- 5.2. Be extremely careful when handling the calibration mass foil set. Contact Thermo Electron Corporation for cleaning instructions. Do not wipe the foil window. Do not touch the foil window with your fingers. See Section 9.4.2 for more details.
- 5.3. Power interruption can cause the instrument's display negative mass and concentration values as the instrument comes back on. To prevent this from occurring as the result of power interruption the following should be done.
 - 5.3.1. Set switch 34 on the back of the instrument (Fig. 2 of the IM, page 13) to "HV buffer". This will maintain a voltage across the detector during any power outage, and minimize measurement loss.
 - 5.3.2. Use an uninterruptible power supply with battery backup (SOP U6) to prevent damage and data loss in case of power failure.
 - 5.3.3. Furthermore, a lightning arrestor will protect the entire on-farm instrument shelter (SOP U1) and secondary surge suppressors will protect the PC and the instruments from electrical surges.
- 5.4. If the heated sampling tube is being used, allow the instrument to equilibrate to room temperature (~ 1 h) before beginning the calibration procedure.
- 5.5. Store and transport the instrument at a temperature in the range -30° to 60°C .
- 5.6. Operate the instrument at a temperature in the range of -20° to 40°C .
- 5.7. Prevent condensation of water in and around the instrument. Avoid situations where the internal temperature is below the dew point of the incoming air. If the instrument is

located inside a conditioned room and the PM₁₀ head is outside, install the sampling tube heater that is available from the instrument manufacturer.

- 5.8. Protect the instrument from precipitation and abrupt changes in temperature.
- 5.9. Some internal components can be damaged by the discharge of static electricity. To avoid damaging internal components, follow these precautions when performing any service procedure:
 - 5.9.1. Wear an antistatic wrist strap that is properly connected to earth ground (note that when the analyzer is unplugged, the chassis is not grounded).
 - 5.9.2. If an antistatic wrist strap is not available, be sure to touch a grounded metal object before touching any internal components.
 - 5.9.3. Handle all printed circuit boards by the edges.
 - 5.9.4. Carefully observe any other instructions for each procedure in the IM.
- 5.10. Do not dismantle, remove or tamper with the C14 radioactive source. It will never be necessary for field personnel to adjust, replace or touch the C14 source. All C14 issues will be handled by the manufacturer.
 - 5.10.1. Maintenance work on the source (e.g. cleaning the measuring section) may only be carried out by an individual trained by Thermo Electron Corporation with suitable authorization.

6. Interferences

- 6.1. Water may collect on the filter tape in humid or rainy climates, causing artificially high mass readings. In hot, humid environments where the instrument is housed in an air-conditioned shelter, heat the inlet tube within the shelter to avoid condensation. In rainy climates, use the instrument's inlet tube heater to ensure that any water drawn into the unit is in the vapor phase as it passes through the filter.
 - 6.1.1. Set the heater at the lowest effective temperature to avoid volatilizing any semivolatile compounds in the PM₁₀. A brief accumulation of water on the filter tape that subsequently evaporates as drier air is pulled through the instrument will cause erroneous short-term readings, but will not adversely affect the 24-h average value
- 6.2. Because the instrument compensates for fluctuations in temperature, the ambient air stream does not need to be heated to a particular standard temperature, as long as it is within the operating range (-20° to 40°C), although heating may still be necessary for moisture control (Section 6.1). Heating the ambient air stream can also eliminate some semivolatile chemicals, leading to inaccurate mass measurements. .

7. Personnel Qualifications

- 7.1. Personnel must be trained in the use of the instrument before initiating the procedure. Training requires about four hours.
- 7.2. Each analyst must read and understand the entire IM and this SOP before operating the instrument.
- 7.3. Personnel should also be trained to interpret the analyzer output signal, set up the internal data logger, and interpret the selected logged parameters.

8. Equipment and Supplies

- 8.1. Method EQPM-1102-150 requires that the C-14 Beta monitor be installed with the following Thermo Electron hardware:
 - 8.1.1. C-14 Beta monitor (Series FH62C14 Continuous Ambient Particulate Monitor)
 - 8.1.2. 10 μ m omni-directional inlet operated at 16.67 L/min (e.g., Model SA246B or as specified in 40 CFR 50, Appendix L)
 - 8.1.3. Sample tube with optional heater
 - 8.1.4. Inlet to sample tube connector
 - 8.1.5. Vertical stabilization of sample tube (if necessary)
 - 8.1.6. Vacuum pump with hose and speed control harness
- 8.2. Keep the following tools, supplies, and spare parts at each remote site:
 - 8.2.1. Glass fiber filter sample tape GF#10, Part No. FH111
 - 8.2.2. Weatherproof silicone caulk or sealant
 - 8.2.3. Certified NIST-traceable flow meter capable of measuring 16.7 L/min
 - 8.2.4. Certified NIST-traceable temperature and pressure sensor
 - 8.2.5. Adhesive tape (necessary after calibration to fix the filter tape to the take-up reel)
- 8.3. Keep the following spare parts in a central stock at PAAQL:
 - 8.3.1. Glass fiber filter sample tape GF#10, Part No. FH111
 - 8.3.2. Pump, Part No. FH180.
- 8.4. Accessories for airflow check or audit:
 - 8.4.1. NIST-traceable volumetric Flow Transfer Standard (FTS)
 - 8.4.2. Unit Coupling: Part No.FH113
 - 8.4.3. 5" Unit Downtube: Part No.FH10819.
 - 8.4.4. Sample Inlet Adapter: Part No.FH113C
 - 8.4.5. Tubing to connect the FTS to the sample inlet adapter

9. Procedure

- 9.1. Instrument setup
 - 9.1.1. Carefully check analyzer and converter for visible defects or damage.
 - 9.1.2. Analog output settings
 - 9.1.2.1. Verify or modify the configuration following the procedure in the IM (Section 2.2.1, page 20) which refers to the Main Circuit Board (Fig. 17 on page 128 of the IM). The same information about pin connections for voltage output or current loop is in Section 10.1.1 of the IM (page 126). Information about the Analog Output Instrument codes is in Table 1, Section 3.9.2 of the IM.
 - 9.1.2.2. The default analog output from the C14 Beta monitor is 0-10 V or 0-20 mA, provided by the status signals and analog output I/O connector (page 13 of the IM). Select between 0-10 V and 0-20 mA analog output according to the DAQ hardware configuration (SOP B1). A summary of the procedure for switching between the 0-10 V and 0-20 mA output options follows.
 - 9.1.2.2.1. Turn the power off, and unplug the instrument.
 - 9.1.2.2.2. Remove the upper, rear panel of the sampler prior to barn installation, so that the Main Circuit Board jumpers can be set or verified.

- 9.1.2.2.3. If there are dummy (temperature) sensor jumpers labeled T1 and T4 installed, remove them in favor of the 2 sensor cables from the rear plug labeled “sensors”.
- 9.1.2.2.4. Check the output configuration terminals, X24 and X10. One is for the rear 25-pin I/O port, and one is for the front face I/O port. These diagrams illustrate the voltage output and current output jumper settings. Both I/O ports should be set up alike, in the Volt Output setting. Note the correct jumper setting:



Figure 3. Jumpers installation for setting up the analog output.

9.1.3. Connections and startup

- 9.1.3.1. Refer to Section 2.2 of the IM (page 18) for connection and startup instructions, including steps to activate external temperature sensors and specify data collection output type.
- 9.1.3.2. Mount the sampler to a rack or shelf inside the OFIS, and attach the inlet tube, which will go through a specially designed flange or collar on the OFIS ceiling. Make sure that the vertical position of the sampler in the rack will allow the sample tube to fit within the ceiling flange once installed.
- 9.1.3.3. Install the waterproof flange on the roof at this time. Install the heated inlet carefully, so that that the bottom of the inlet tube just reaches the instrument inlet. When properly installed, the heated inlet tube will hang vertically over the top of, and perpendicular to, the top of the sampler at the inlet point, where a mating collar is then secured into place.
- 9.1.3.4. Pass the ambient temperature sensor, whose lead protrudes from the end of the heated inlet tube under the roof flange, through the roof flange, and mount it inside the sun shield, which is clamped onto the outside inlet tube that supports the PM₁₀ head and cyclone.
- 9.1.3.5. Connect the 9-pin connection (attached to cables that protrude from the lower end of the inlet tube) to the rear panel of the sampler at the “Sensors” mating socket.
- 9.1.3.6. Connect the inlet tube heater power supply to the large, metallic clamps on the tube using Allen screws. Use only one screw per clamp for power lead connections. One heavy supply output lead goes to one clamp, and the other supply output lead goes to the other clamp. Connect the power supply input to the rear panel of the sampler, as marked.
- 9.1.3.7. Plug the pump directly to AC power, and to the marked electrical and pneumatic connections on the rear of the sampler.
- 9.1.3.8. Turn on instrument. The detector needs 3 to 4 h for stabilization, after the power is first turned on. The instrument will generally display negative mass and concentration values during this time.

- 9.1.3.9. If the filter tape is already installed, advance it to the next position (Section 9.2.4.1.3).
- 9.1.3.10. If not already installed, attach and install a roll of approved filter media, intended for this instrument, to the left side spindle, unwinding clockwise. Use the controls described under the Maintenance menu to open the filter head. Bring the sample media up and over the transport sensor wheel (resembling a tension roller), and pass the media through the sample head left to right. Use a piece of office or masking tape to secure the tape head to the take-up spool.
- 9.1.3.11. Data collection is accomplished by wiring a male 25-pin I/O connection as follows: Pin 12 = 10V+ out and Pin 13 = 10V- out. Run this data collection wire pair to an input channel of the DAQ system for real-time data collection.
- 9.2. Operation of the keypad or screen
- 9.2.1. Once the sampler has been set up, certain operating parameters will be set or verified for proper operation and data collection. It is important to understand the operation of the keypad or screen itself, to conduct these operations.
- 9.2.2. Verify that the C14 Beta monitor's parameters are set to the following values:
- 9.2.2.1. Operated for 24-h average measurements
 - 9.2.2.2. Flow rate of 1000 L/hr (16.67 L/min)
 - 9.2.2.3. Daily automatic filter change (1,440-min cycle time)
 - 9.2.2.4. Automatic filter change when flow rate <950 L/h
 - 9.2.2.5. Automatic filter change when sampled filter spot mass is > 1,500 mg
 - 9.2.2.6. Factory default calculation mode settings (60 min LR time, 10% STDEV, 33 s RC mass filter)
- 9.2.3. The sampler has a view screen (Fig. 4), with options at the bottom of the screen which may be affected by the keypad immediately beneath it. The softkeys correspond to the functions just above them. Fig. 4 shows a different name for each of the two left soft keys. The first and second softkeys from the left correspond to one of the two functions shown above them, depending on which specific screen is selected.



Figure 4. View screen for data readout and for input of operating parameters.

- 9.2.3.1. The “Next” key moves to the next screen in the rotation of selectable options. Executing this button saves the information present in the screen at that point.
- 9.2.3.2. The “Back” selection moves to the prior screen in the rotation of selectable options. NOTE – going “Back” will fail to save whatever parameters were changed on a given screen. Only going “Next” will save entries to the screen.
- 9.2.3.3. “Yes” moves to a sub-menu under a given heading, or executes actions which are described on the display, or switches on parameter flags (as appropriate).
- 9.2.3.4. “No” switches off parameter flags.
- 9.2.3.5. “+” and “-” change input values and parameter values.
- 9.2.3.6. These principles of value change apply to all areas of screen operation.
- 9.2.4. The Value Display is usually seen while the sampler is running. It shows the word “Normal”, a 4-digit status line, a 2-digit error code line, and the present active sample concentration in $\mu\text{g}/\text{m}^3$. Under normal running conditions, the three indicator lights to the right of the keypad/screen will show:
 - 9.2.4.1. Green LED “Ready” is on.
 - 9.2.4.2. Yellow LED “Status” is off.
 - 9.2.4.3. Red LED “Status” is off.
 - 9.2.4.4. **Pressing the “Next” key once** from this view will show concentration, mass, and airflow rate.
 - 9.2.4.5. **Pressing “Next” a second time** will show the airflow rate at the sample inlet, and the flow rate adjusted for standard temp (25°C) and pressure (760 mm/Hg).
 - 9.2.4.6. **Pressing “Next” a third time** will show 30-min, 3-h, and 24-h average PM concentrations.
 - 9.2.4.7. **Pressing “Next” a fourth time** will show the Radon content in the current sample.
 - 9.2.4.8. **Pressing “Next” a fifth time** will show status or error codes, with a “Text” option displayed; choosing “Text” will give information about the error.
 - 9.2.4.9. **Continuing with “Next”** will offer the chance to set date and time.
- 9.2.5. Unlocking the screen
 - 9.2.5.1. In order to make changes to menu items, the keyboard must be unlocked. To unlock the screen, **go to any one of the other view screens - Operations, Maintenance, or Calibration - by pressing the button under the word “Yes”**. Once there, the question “Keys Locked?” will be seen [“On Line = Yes” is displayed]. Press the “No” button to affirm that the keypad is not to remain locked [“On Line = No” is displayed]. The following becomes true when the keypad is unlocked under any menu screen:
 - 9.2.5.1.1. The yellow Status LED will come on as a reminder that the machine is not in normal operation.
 - 9.2.5.1.2. The keyboard is free, so that all parameters can be changed with the “+” and “-“ keys.
 - 9.2.5.1.3. A manual filter change can be initiated by pressing the “FC+Z” keys.
 - 9.2.5.1.4. Serial and parallel interfaces are disabled, though control signals and other electronic inputs are unaffected if such were to become in use.

- 9.2.6. Further explanations of how this instrument operates, in all following sections, will include references to the sampler view screen. Options at the bottom of the screen represent changes that may be affected by the keypad immediately beneath it.
- 9.2.7. The Operations Menu allows the operator to set chiefly interface issues, such as analog outputs, baud rates (if serial communications are used), etc.
- 9.2.7.1. Unlock the keypad, and then use the “Next” key to move through the following parameter choices, and make the selection or use the “+” or “-” or specific words presented, depending upon the choices available, to adjust the settings as needed.
- 9.2.7.1.1. Press the button beneath the COM# displayed to toggle between COMM1 or COMM2. Press “Yes” to save and continue to the next option.
- 9.2.7.1.2. This section of variables allows the user to specify a print format for the capture of near real-time data through the COMM port, and to specify output cycle intervals. The IM shows example formats on page 112.
- 9.2.7.2. The screens labeled Analog Outputs allow the operator to configure output signals to match parameters used by the data logger of choice at the installation location. Press “Yes” to check or change settings.
- 9.2.7.2.1. The first option under this heading determines the voltage output seen by the logger under a zero reading on the instrument. Note that the instrument should have already been set up to deliver voltage output, not milliamp output; the screen will still show references to “mA” where 0 mA = 0 V, and 20 mA = 10 V. For ESC data logger use, set the Output Zero to correspond to 0 mA.
- 9.2.7.2.2. The second option selects which output will be chosen from Section 3.9, Table 1, on page 40 of the IM. Choose Selection 10 (mass concentration in $\mu\text{g}/\text{m}^3$).
- 9.2.7.2.3. The third option under this heading allows setting of the span value desired for mass concentration. Again, the screen will read 20 mA for what is, in fact, a 10-V span output.
- 9.2.7.3. The Filter Change screen parameters which follow specifies under which conditions a filter change will occur. Press “Yes” to check or change settings.
- 9.2.7.3.1. The first settable trigger is for mass concentration; set this for 1500 $\mu\text{g}/\text{m}^3$. If a sample should exceed this limit, an extra filter change will occur.
- 9.2.7.3.2. The second variable is for airflow; if flow falls below this set level a filter change will occur. 950 L/h is the normal setting.
- 9.2.7.3.3. The next setting will cause a filter change after a preset period of time since the last one. Set this cycle to 1440 min (one change per day).
- 9.2.7.3.4. Finally, select the hour of the above filter change. For midnight, enter “24:00”, since “00:00” represents NO regular filter change interval for this instrument.
- 9.2.7.4. The Heater and Air Flow rate screen determines the operating temperature of the condensation-preventing heated inlet tube, and the sample air flow rate. Press “Yes” to check or change settings.
- 9.2.7.4.1. The first screen displayed upon entering this section shows the percent of heater regulator effort (i.e. 567 = 56.7%), the nominal set point in $^{\circ}\text{C}$ for the inlet, and the present, measured inlet temperature in $^{\circ}\text{C}$. Enable the ability to change the set point by pressing the NOMIN key.

- 9.2.7.4.1.1. Use the “+” and “-“ keys to select an appropriate operating temperature. A range around 40°C will be common. Press “NEXT” to save and continue.
- 9.2.7.4.2. The next screen will show Air Flow regulatory status. As with inlet temperature, this will display regulator effort percent (i.e. 638 = 63.8%), a nominal set point, and the actual flow as measured.
- 9.2.7.4.2.1. Enable the ability to change the set point by pressing the NOMIN key.
- 9.2.7.4.2.2. Use the “+” and “-“ keys to select the standard air flow of 1000 L/h. Press “NEXT” to save and continue.
- 9.2.7.5. The Concentration Thresholds, labeled “Limits Concentration, is the next settable screen. Press “Yes” to check or change settings, and “Next” to save and go past.
- 9.2.7.5.1. Limit 1 is the level at which the yellow status LED will be activated and a display of STAT 0100 will be shown.
- 9.2.7.5.2. Limit 2 determines the level at which a display of STAT 0200 will be shown.
- 9.2.7.5.3. A display of STAT 0300 will be seen if both Limit 1 and Limit 2 are exceeded.
- 9.2.7.6. A language screen allows adjustment for operation in various countries.
- 9.3. Instrument operation
- 9.3.1. Routine operation of the instrument involves allowing the sampler to run in a stable temperature environment around 25°C, changing filter media approximately once per a year (depending upon consumption), periodic adjustment of inlet tube temperature as required, calibration, and following trends in sample data or a break in the usual pattern of collection for possible anomalies.
- 9.3.2. Tape handling and pump operation are operated through the Maintenance Menu. Unlock the keypad (Section 9.2.4), and use the “Next” key to move through the available options, and make the selection or use the “+” or “-” or specific words presented. Use “Next” to initiate actions and to go to the next step, as needed.
- 9.3.2.1. Pump operation is the first control available. It is necessary to start and stop the pump during various phases of use. DO NOT simply pull the pump control plug from the rear of the sampler to stop the pump.
- 9.3.2.2. The next three options allow one to open the sample head, move the filter strip manually, and close the head. The head is opened during set-up, and for calibrations. Closing the head will begin an automatic zero reference check.
- 9.3.2.3. Strictly follow the IM when performing any further functions under Maintenance, as they do not in any way reflect routine operation or maintenance of the instrument.
- 9.4. Instrument Calibration
- 9.4.1. Enable Calibration Mode. Follow procedure in Section 9.2.5.1 to access this mode.

<p>CALIBRATION ***** YES BACK NEXT</p>

- 9.4.1.1. In this mode the particulate mass, the air flow rate, the temperature, and pressure sensors are calibrated. Correct calibration of these parameters is necessary to accurately record the particulate concentration

CALIBRATION
KEYS LOCKED ?
(ON LINE) NO NOYES
BACK NEXT

- 9.4.1.2. If the keys are locked, the following parameters cannot be modified. Press NO to unlock the keys

CALIBRATION
CHANGE NO.: 12345
02-07-23 13:30
BACK NEXT

- 9.4.1.3. The running number (date + time) of the parameter change or recalibration is indicated. With each parameter or calibration change the change number and date (yy-dd-mm) are entered. Press NEXT.

CALIBRATION:
CALIB. CODE:
0
- + BACK NEXT

- 9.4.1.4. Enter the code "147" in this screen: This is necessary to protect sensitive calibration parameters. Other passwords are used for special setups.

- 9.4.1.5. Note: The calibration menu is locked if the calibration menu is inactive for 4 min.

Wrong code:

CALIBRATION
CALIBR. LOCKED !
12345
- + BACK NEXT

Valid code:

CALIBRATION
CALIBRATION FREE !
147
- + BACK NEXT

- 9.4.1.6. After the correct password is entered, the status bit "calibration enabled" is set.

CALIBRATION
AMPLIFICATION:
7250 µg
_ + BACK NEXT

- 9.4.1.7. The mass calibration value is indicated, and can be entered. This mass - calibration factor [µg] is determined from the instrument during the reference-foil calibration. It converts the detector count rate into the mass value. The keys "-" and "+" appear only if the calibration is enabled, and these are only used for a fine tune adjustment of the Span (e.g., seasonal adjustments if necessary).

- 9.4.1.8. Record the calibration values of the temperature sensor, pressure sensor, flow rate, and mass foil calibration in the **"Thermo C-14 Beta Sampler Calibration Form"**. (Appendix A).

9.4.2. Mass Foil Calibration

- 9.4.2.1. The foil calibration procedure is a user-interactive mode, and should be performed annually, or as stated in the QAPP, or after service to the measurement chamber. Follow all instructions displayed on the screen. The complete process is automatically directed by the C14 BETA firmware. The complete procedure lasts approx. 20-30 min. If necessary, stop the procedure using the ABORT key.
- 9.4.2.2. **WARNING:** Take very good care of the calibration foil set. Contact Thermo Electron Corporation for cleaning instructions. Do not wipe the foil window. Do not touch the foil window with your fingers.
- 9.4.2.3. Perform calibration only with the heater off and the unit thermally equilibrated.

CALIBRATION
FOIL CALIBRATION
PROCEDURE ?
YES BACK NEXT

- 9.4.2.4. Press YES to access the calibration mode using the reference mass foils. Follow the instruction displayed on the screen.

CALIBRATION PROCEDURE
SPAN FOIL VALUE:
1442 µg
- + BACK NEXT

- 9.4.2.5. Enter the SPAN foil value (choose the mid-range foil for 0-5,000 µg/m³ range or maximum value foil for 0-10,000 µg/m³ range) Mid-Range Span is to be used for USEPA Equivalency. Press NEXT

CALIBRATION PROCEDURE
HEAD OPENS !
PLEASE WAIT!
ABORT

- 9.4.2.6. The sample/measurement chamber opens. This screen is displayed while the measuring head opens (about 30 s).

CALIBRATION PROCEDURE
INSERT FOIL HOLDER !
ABORT NEXT

- 9.4.2.7. Cut filter tape from take-up reel and pull it out. Then insert the foil holder frame from the left into open gap. Note that the word “oben” on the foil holder should be facing “up”. (oben = up) Press NEXT.

CALIBRATION PROCEDURE
HEAD CLOSES
PLEASE WAIT!

- 9.4.2.8. This screen is displayed while the measuring head closes (~ 30 s).

CALIBRATION PROCEDURE
INSERT ZERO FOIL!

ABORT **NEXT**

9.4.2.9. Insert the zero foil (Nullfolie) with the word “unten” facing down. Close the front instrument cover. Press NEXT. The zero has now begun.

CALIBRATION PRO 27%
AUTOZERO ACTIVE
115 4 12*
ABORT TIMER:355

9.4.2.10. The offset and mass indication during the mass auto zero lasts ~ 7.5 min. Wait until the timer count down is ready. If offset is > +500 %, then the procedure stops. Contact Thermo Electron Corporation if this occurs.

CALIBRATION PRO
TAKE OUT FOIL

ABORT **NEXT**

9.4.2.11. Once the Zero Foil calibration is complete, take out the zero foil and place in the protective case. Press NEXT.

CALIBRATION PRO
INSERT SPAN FOIL.

ABORT **NEXT**

9.4.2.12. Insert the chosen SPAN foil with the word “unten” facing down. Close the front instrument cover. Press NEXT.

CALIBRATION PRO
CALIBRATION ACTIVE
79 1515 1451*

ABORT

9.4.2.13. Wait until the calibration timer countdown has been completed (10-15 min).

CALIBRATION PRO
RATE 0 (1/s) mass(μ g) 2 1515
1451*

ABORT

9.4.2.14. This screen is displayed during the Span Calibration, and shows the count rate and mass value.

AMPLIFICATION
OLD: 7505 NEW: 7489
NEW VALUE ?

NO **YES** BACK **NEXT**

9.4.2.15. The old and new amplification values are shown when completed. Press "YES" to accept the new calibration value

9.4.2.16. Press "NO" to decline the new calibration value

9.4.2.17. Press "BACK" to repeat the foil calibration procedure. Then Press NEXT.

CALIBRATION PRO
HEAD OPENS
PLEASE WAIT

9.4.2.18. By Pressing "ABORT" during the calibration procedure, this screen will be displayed.

CALIBRATION PRO
TAKE OUT FOIL
INSERT FILTER STRIP
NEXT

9.4.2.19. At the end of the calibration, take out the foil and then the foil holder. Insert the filter tape and adhere it to the take-up reel. Press NEXT.

CALIBRATION PRO
WRITE PRESET VALUES
INTO EEPROM
YES BACK NEXT

9.4.2.20. To write the new calibration values into memory, Press YES. The parameters and calibration data are now stored into the EEPROM. An automatic reload of this calibration parameter will happen after a cycling of the power.

9.4.3. Airflow Rate Verification

- 9.4.3.1. As per 40CFR, Part 50, Appendix L, Section 9.2.5, the flow rate of the Model C14 Beta should be verified. Check the flow rate at least quarterly, or as stated in the QAPP.
- 9.4.3.2. Complete the temperature and pressure verification tests conducting this Flow Rate Verification.
- 9.4.3.3. Prepare the equipment and parts listed in Section 8.4.
- 9.4.3.4. Attach the small sample tube adapter to the Model C14 Beta inlet.
- 9.4.3.5. Attach the inlet adapter to the sample tube. Depending upon what NIST-traceable volumetric flow transfer standard (FTS) is being used, the FTS may either be attached now, or a flow audit adapter may be added to accommodate any flexible tubing that the FTS may require.
- 9.4.3.6. Allow 60 s for flow to stabilize.
- 9.4.3.7. Section 9.2.4.5 describes the procedure to display the airflow rate at the inlet, and as the airflow rate corrected to standard conditions (25°C and 1 atm). Compare the value obtained with the audit flow meter with the air flow rate at the inlet value displayed by the monitor.
- 9.4.3.8. Record the Model C14 Beta Volumetric Flow Rate as Q_m and the FTS flow rate as Q_i . Use units of L/h for both.
- 9.4.3.9. Take three (3) readings each of Q_m and Q_i , and average the respective values.
- 9.4.3.10. Use the following equation to calculate the percent difference (%D):

$$\%D = (Q_i - Q_m) / Q_i * 100 \quad (9.1)$$

- 9.4.3.11. If the %D is within $\pm 4\%$, then the volumetric flow rate is acceptable.

9.4.3.12. If the %D of the volumetric flow rate is more than $\pm 4\%$:

9.4.3.12.1. If the test is an acceptance verification test conducted after unpacking a new instrument, contact Thermo Electron's Technical Support, or your local sales representative.

9.4.3.12.2. Conduct a calibration test (Section 9.4.4) if it is a test other than the initial verification test.

9.4.3.13. Volumetric flow rate calibration is covered in Section 9.4.4.

9.4.4. Air Flow Rate Calibration

9.4.4.1. For a flow rate calibration, use a NIST-traceable volumetric flow calibration device (e.g. flowmeter). Calibrate all temperature and barometric pressure sensors before conducting the air flow rate calibration. Perform calibration once per year, or as directed in the QAPP. Perform calibration after the instrument has been moved from its location, after any major repair or maintenance, or after poor audit results.

<p>CALIBRATION AIR FLOW:</p> <p>YES BACK NEXT</p>
--

9.4.4.2. Press YES to begin the airflow rate calibration.

<p>CALIBRATION AIR FLOW:</p> <p>MEAS.: 999 l/h CAL BACK NEXT</p>

9.4.4.3. The volumetric flow rate is initially indicated as measured by the C14 BETA. Press CAL to change the air flow rate calibration coefficient. Thereafter, enter the actual air flow rate measured by a reference instrument and press NEXT. Allow 30 s between indicated airflow adjustments. Repeat as necessary until corresponding reference and C14 BETA flow rate values agree within 2%.

<p>CALIBRATION VOLUME REFERENCE :OPERA SET BACK NEXT</p>

9.4.4.4. The conditions (temperature and pressure) of the air flow rate used for the calculation of the concentration can be set here. Press SET to toggle between the following options:

OPERA	volumetric conditions
273° K	273°K , 1013 hPa
293° K	293°K , 1013 hPa

9.4.5. Temperature and Pressure Sensor Calibration

9.4.5.1. The calibration of temperature and pressure sensors, indicated by a reference unit, is entered here. The instrument starts an automatic adjustment after each calibration entry. Values are stored in battery-buffered RAM with check sum.

CALIBRATION
TEMPERATURE- AND
PRESSURE SENSORS
YES BACK NEXT

9.4.5.2. Press YES to access the submenu for calibrating the temperature and pressure sensors.

CALIBRATION OF SENS.
TEMP. PM 10 HEAD
MEAS.: 23 °C
CAL BACK NEXT

9.4.5.3. The ambient temperature sensor (T1) is indicated in this screen. Press CAL to change its value. Enter the measured value of a reference thermometer using the ± keys.

CALIBRATION OF SENS.
TEMP. PM 10 HEAD
MEAS.: 23 °C
- + BACK NEXT

9.4.5.4. The adjustment procedure lasts about 5 to 30 s. During this time, a "K" flashes in the first line on the right.

9.4.5.5. When this procedure is finished one of the following texts appear on the screen:

CALIBRATION OF SENS.
CALIBRATION O.K.
MEAS.: 25 °C
CAL BACK NEXT

CALIBRATION OF SENS.
CALIBR NOT POSSIBLE
MEAS.: 23 °C
BACK NEXT

9.4.5.6. If the message "CALIBR NOT POSSIBLE" appears, then the new calibration value cannot be reached within the limits of the adjustment range. Check the entry that was made, check for a loose connection on the T sensor cable, or for a frayed wire.

9.4.5.7. Use the same calibration procedure for the following sensors:

CALIBRATION OF SENS.
TEMP. UPPER HEAD
MEAS.: 26 °C
CAL BACK NEXT

9.4.5.8. T2: Temperature sensor over the filter tape

CALIBRATION OF SENS.
TEMP. LOWER HEAD
MEAS.: 25 °C
CAL BACK NEXT

9.4.5.9. T3: Temperature sensor under the filter tape

CALIBRATION OF SENS.
TEMP.INLET HEATER
MEAS.: 40 °C
CAL BACK NEXT

9.4.5.9.1. T4: Temperature sensor at the tube wall of the heated sample tube (optional)

CALIBRATION OF SENS.
AIR PRESS(ABSOLUTE)
MEAS.: 958 hPa
CAL BACK NEXT

9.4.5.10. The value of the ambient pressure sensor (P3) is indicated here. Press CAL to change its calibration (hPa = in Hg / 33.7685) Enter the value obtained from a reference barometer, using the + or - key. Press NEXT to finish the data input. Allow ~30 s for the adjustment procedure.

9.4.5.11. After calibrating the barometric pressure sensor (P3), it is usually not necessary to calibrate the P1 and P2 sensors. If these sensors are operating in the 3500-4500 Pa and 80-100 hPa range, respectively, then calibration is not needed. If annual calibration is needed, remove the top cover of the C14 BETA to access the pressure line upstream of the orifice.

CALIBRATION OF SENS.
ORIFICE PRES(ZERO)
MEAS.: 0 Pa
CAL BACK NEXT

9.4.5.12. The value of the orifice pressure sensor (P1) is indicated here. Press CAL to change its calibration when the pump is switched off. Enter zero (0) with the unit at rest. Press NEXT to finish the data input. Allow ~30 s for the adjustment procedure.

CALIBRATION OF SENS.
ORIFICE PRESS(SPAN)
MEAS.: 5098 Pa
CAL BACK NEXT

9.4.5.13. The value of the orifice pressure sensor (P1) is indicated here. Apply a pressure of 50 hPa (5000 Pa) on the upstream orifice pressure line. Press CAL to change its calibration of the slope (SPAN). Measure the exact value using a reference instrument and enter this value using ± keys. Press NEXT to finish the data input.

CALIBRATION OF SENS.
HEAD(ZERO): MEAS.: 0
hPa
CAL BACK NEXT

9.4.5.14. The value of the vacuum/pressure (under filter tape) sensor (P2) is indicated here. Press CAL to change its offset (zero point). Switch off the pump and enter the zero value. Press NEXT to finish the data input. The adjustment procedure starts.

CALIBRATION OF SENS.
HEAD(SPAN): MEAS.: 210
hPa
CAL BACK NEXT

9.4.5.15. The value of the vacuum/pressure sensor (P2) is indicated here. Apply a vacuum of about 200 hPa on the upstream orifice pressure line. Measure the exact value using a reference instrument and enter this value using + or – key. Press CAL to change its calibration of the slope (SPAN).

WRITE PRESET VALUES
INTO EEPROM ?
YES BACK NEXT

9.4.5.16. All parameters, date of inserting the source and calibration data are written in the EEPROM. A reload of these data happens if the power is cycled and an error in the battery-buffered RAM is triggered or if through an explicit keypad command in the maintenance menu a successful writing or read command was applied.

EEPROM WRITE O.K.

EEPROM ERROR !

BACK NEXT

BACK NEXT

9.5. Calibration of the C14 BETA

9.5.1. Mass Calibration

9.5.1.1. Do not touch the calibration foil windows inside the holder.

9.5.1.2. Note: If the heated sampling tube is being used, the instrument should equilibrate to room temperature before the calibration procedure begins (~ 1 hr).

9.5.1.3. The C14 BETA has no front access to the filter tape. Therefore, the filter tape must be cut near the measuring head and removed from the side of the measurement head. After the calibration procedure, the filter tape must be inserted and fixed to the take-up reel using an adhesive tape.

9.5.1.4. The calibration procedure uses an internal menu operation. The menu operation is comprehensively described in Section 4.2.4.1 of the IM (Mass Foil Calibration Procedure, page 65),

9.5.1.4.1. Access the Calibration submenu KEYS LOCKED ? and confirm with key NO.

9.5.1.4.2. Access the Calibration submenu CALIBR. CODE and enter the release code 147.

9.5.1.4.3. Activate the foil calibration procedure via the Calibration, submenu FOIL CALIBRATION PROCEDURE ? and confirm with YES.

9.5.1.4.4. Enter SPAN Mass Foil Value (~ 1,400 µg, see the foil kit imprint), and press NEXT.

9.5.1.4.5. The measurement chamber will open. Remove the filter tape from the particulate sampling position and insert the guide bar into the filter gap on the left with the word “oben” facing up. Press NEXT.

9.5.1.4.6. Insert the zero foil from the left with the word “unten” facing down. Close the front monitor window.

9.5.1.4.7. Press NEXT. The filter spot is closed and a zero adjustment of the mass is initiated (3-5 min). Offset and mass value are indicated.

9.5.1.4.8. Next, the order appears in the display to take out the zero foil and to insert the SPAN foil. Confirm by pressing NEXT.

- 9.5.1.4.9. The SPAN adjustment runs approximately 5-10 min, until the measurement value stabilizes itself. The actual mass value, as well as the remaining measurement time, is indicated.
 - 9.5.1.4.10. Next, the old and new calibration factors FCAL are indicated. Use the YES key to accept the new calibration factor is accepted, or NO to discard it.
 - 9.5.1.4.11. The measurement value of the SPAN foil is indicated and can be compared with the imprinted value. Confirm with NEXT.
 - 9.5.1.4.12. The measurement chamber is opened again. Take out SPAN foil and guide bar and insert filter tape and adhere it to the take-up reel. With the NEXT key, a filter change is triggered and the measurement operation starts again.
- 9.5.2. Mass Validation
- 9.5.2.1. Use the same or an independent set of mass transfer standard foils to conduct an audit or verification of the mass calibration. Validate using the following steps. It is best to understand the above mass calibration procedure first. Within the Maintenance Menu, perform the following steps:
 - 9.5.2.1.1. Enable the keys, switch off pump, open the head, and remove the filter tape
 - 9.5.2.1.2. Insert the foil holder. Carefully insert and align the null foil
 - 9.5.2.1.3. In the Maintenance Menu → Close the Head and Zero.
 - 9.5.2.1.4. Wait 8 min for the zeroing cycle to finish.
 - 9.5.2.1.5. After zeroing cycle is complete (as seen on the front display), remove the null foil and insert the mid-range span foil.
 - 9.5.2.1.6. From the Main Menu, press DISP to obtain a direct reading of the detected mass. Compare with the imprinted foil value; the two should be within 5% of each other. If not, redo the verification. If an error >5% persists, recalibrate.
- 9.5.3. Instrument configuration
- 9.5.3.1. A screen that reads “Configuration ETC” can be found following the Calibration Menu. A release code has to be entered to unlock this screen.
 - 9.5.3.2. Do not change anything under this heading, unless it can be demonstrated that the values used are consistent with USEPA Equivalent Method Designation practices. Reconfiguration during the warranty period will void the instrument warranty.
 - 9.5.3.3. Once the configuration screen has been entered, the following settings should remain as set. Advance to the following screen via “Next” button, in each case:
 - 9.5.3.3.1. Calculation Mode = 0 [zero].
 - 9.5.3.3.2. Regression Time = 60 min.
 - 9.5.3.3.3. Standard Deviation = 10%.
 - 9.5.3.3.4. Efficiency Ea2 = 20.3%.
 - 9.5.3.3.5. Negative Concentration output = Yes.
 - 9.5.3.3.6. RC-Filter for Mass = 33 s.
 - 9.5.3.3.7. Comp. Factor F (R1/R2) = 360% [Do Not Change].
 - 9.5.3.3.8. Version: 86,84,92,EPA = 0 [zero, for std. EPA]
 - 9.5.3.4. If changes are made, a following screen prompts the operator to write preset values into the EEPROM. The usual answer will be “Yes”, in order to save such changes. Proceed to “Next” after doing so.

9.6. Maintenance

9.6.1. Maintenance schedule

Maintenance Schedule Recommended by Manufacturer	Maintenance Procedure	IM Section	SOP Section
1-3 mon.	Check air flow	4.2.4.2	9.4.3
3 mon.	Clean/inspect PM ₁₀ inlet head	5.6	9.6.3
3 mon.	Mass validation with foil set	5.11.3	9.5.2
6-9 mon.	Check/replacement of filter tape	4.2.3	9.6.5
12 mon.	Mass calibration with foil set	4.2.4.1 & 5.11.2	9.4.2
12 mon.	Air flow calibration	4.2.4.2	9.4.4
12 mon.	Replace pump carbon vanes	5.4	9.6.8
2 yrs	Cleaning measurement chamber	Contact: TEC	

9.6.2. Check airflow.

9.6.2.1. Use the procedure described in Section 9.4.3.

9.6.3. Clean and inspect PM₁₀ sampler inlet head

9.6.3.1. Inspect the water collector bottle (located on the sampler inlet head tube) at least every five sampling days (Fig. 5). Remove any accumulated water, clean the interior of the bottle, inspect the seals, and replace the bottle in the holder.

9.6.3.2. Dismantle and clean the sampler inlet once a month, or as specified in the QAPP. Mark each assembly point of the sampler inlet with a pen or pencil to provide reference marks during reassembly.

9.6.3.2.1. Disassemble the sample inlet unit. If the assembly screws are frozen, apply penetrating oil or commercial lubricant to make removal easier. Clean all interior surfaces and the bug screen with a general purpose cleaner or compressed air, paying particular attention to small openings and cracks. Cotton swabs and/or a small brush are helpful. Completely dry all components.

9.6.3.2.2. Check the O-rings for distortion, cracks, fraying, or other problems, and replace as necessary. Apply small amounts of grease to the rings before assembling the unit. Reassemble the unit in accordance with the previously scribed match marks. Take particular care to ensure that all O-rings seals are properly seated, and that all screws are uniformly tightened.

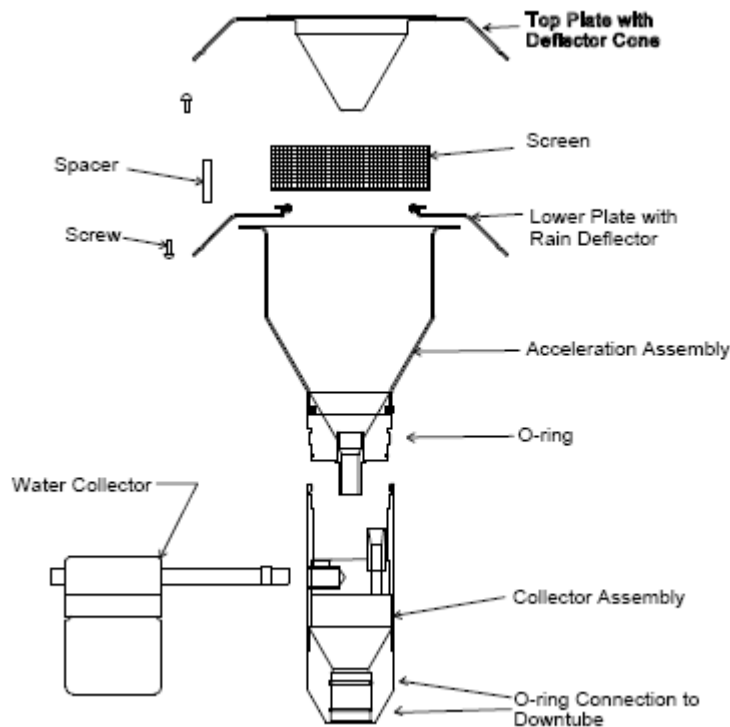


Figure 5. PM₁₀ sampler head.

9.6.4. Mass validation with foil set.

9.6.4.1. Follow the procedure described in Section 9.5.2.

9.6.5. Check/replacement of the Filter Tape

9.6.5.1. As soon as the filter tape on the filter roll is empty, replace the roll.

9.6.5.2. The filter tape is usually replaced annually, but this can also depend on the non-standard operational parameters chosen by the user or the loading per filter spot. In high-concentration areas, more frequent filter changes can occur due to higher loading. As a basis for estimation, note that 42 m of tape per roll is sufficient for approximately 800-900 single measurements. Due to the uncertainty of filter tape life in high-concentration areas, check the filter tape every 6-9 mos.

9.6.5.3. To change the filter tape, follow the instructions in Section 4.2.3 of the IM, under the Maintenance Menu, and Section 9.1.3.10 of this SOP.

9.6.6. Mass calibration with foil set

9.6.6.1. Follow procedure in Section 9.4.2.

9.6.7. Air flow calibration

9.6.7.1. Follow procedure in Section 9.4.4.

9.6.8. Carbon Pump Vane Replacement

9.6.8.1. Vane replacement must be done outdoors. Failure to do so may introduce carbon particles into the air and damage electronic equipment.

9.6.8.2. Before servicing, the vacuum pump must be switched off and secured from any accidental start-up.

- 9.6.8.3. In order to replace the vanes, remove the silencer housing (1), the silencer diaphragms (2), the distance ring (3), and the cylinder cover (4) (Fig. 6).

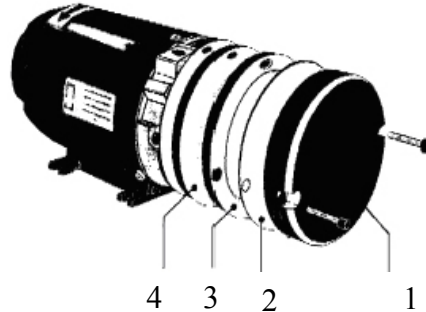


Figure 6. Pump Parts Location.

- 9.6.8.4. Blow carbon particulate from vane cavity (Fig. 7) with an air hose.
- 9.6.8.5. **WARNING:** Avoid breathing in carbon particulate. Used an approved particle filter or respirator.
- 9.6.8.6. When installing new vanes, be sure that the beveled side points outwards.
- 9.6.8.7. Reassemble the parts in the reverse order.

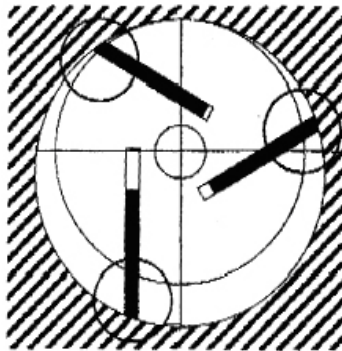


Figure 7. Carbon Vane Installation.

- 9.6.9. Clean the measurement chamber. Contact TEC service department.
- 9.7. Record all maintenance on the “Monthly Maintenance Checks” form (Appendix B of this SOP).
- 9.8. Troubleshooting: Section 12 of the IM (page 132) presents a list of errors, potential causes, and appropriate corrective actions.

10. Data and Records Management

- 10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet that is designated for this method. Supplement this electronic record with a bound record

book designated for the method, which should also contain bound copies of the electronic record.

- 10.2. Manage all data according to SOP B5.
- 10.3. Document all data and information on field data sheets, and site logbooks with permanent ink, or in electronic field notes.
- 10.4. Overstrike errors in writing with a single line; initial and date any correction on the hard copy notes.
- 10.5. Use the “Thermo C14 Beta Sampler Calibration Form” (Appendix A) to record the calibration values before transferring them to the electronic form in the daily notes.
- 10.6. Use the “Monthly Maintenance Checks – Thermo Electron FH 62C14 (Beta) Continuous Ambient Particulate Monitor” form (Appendix B), to record the monthly maintenance activities before transferring the information to the electronic form in the daily notes. Store all monthly maintenance forms on-site, in a three-ring binder that is labeled for this instrument.

11. Quality Control and Quality Assurance

- 11.1. Calibration of mass, temperature, pressure and air flow rate, is performed periodically as described in this SOP (Sections 9.4.2, 9.4.4, 9.4.4, and 9.4.3 respectively), and according to the QAPP.
 - 11.1.1. PM mass: The mass measurements of the C14 BETA instruments are calibrated to a mass transfer standard foil set (Section 9.4.2). These foil sets are calibrated against quartz particulate (SiO_2), in comparison to a calibrated gravimetric mass measurement. The original calibration values are listed in the factory test papers delivered with the instrument.
 - 11.1.1.1. Validate mass calibrations quarterly (Section 9.5.2).
 - 11.1.1.2. Re-calibrate mass measurements annually (Section 9.4.2).
 - 11.1.1.3. Re-calibrate mass measurements (Section 9.4.2) after intrusive maintenance and repair work, and to validate the mass span setting.
 - 11.1.2. Airflow rate
 - 11.1.2.1. Check the measurement at least quarterly (Section 9.6.2).
 - 11.1.2.2. Calibrate (Section 9.4.3) annually with a NIST-traceable volumetric flow transfer standard that is accurate within $\pm 2\%$.
 - 11.1.3. Temperature
 - 11.1.3.1. Calibrate (Section 9.4.5) at least quarterly. Use a NIST-traceable temperature standard accurate within $\pm 0.5^\circ\text{C}$.
 - 11.1.4. Barometric pressure
 - 11.1.4.1. Calibrate pressure sensors (Sections 9.4.5.10-9.4.5.15) at least quarterly. Use a NIST-traceable pressure standard accurate within ± 2 mmHg.
- 11.2. The manufacturer has not developed any recommended leak checking procedures. Do not put the system under high positive or negative pressure; damage to the measurement system could result.
- 11.3. All the NIST traceable calibration standards certifications are to be kept up-to-date.
- 11.4. All AC powered Transfer Standard equipment will be allowed to equilibrate for at least one hour to ambient conditions.

- 11.5. Daily data will be inspected within three days for completeness. Any anomaly will be investigated, first by contacting the site engineer, and then by contacting the site Principal Investigator.

12. References

- 12.1. FH62C14 Instruction Manual.. 2004. Series FH62C14- Continuous Ambient Particulate Monitor Instruction Manual. P/N 100230-00. Thermo Electron Corporation, Franklin, MA. Revision of March 2005.
- 12.2. Delaware Department of Natural Resources and Environmental Control (DNREC). 2003. Standard Operating Procedure C-14 (Beta) sampler for continuous 2.5 μ g Particulate Monitoring. Word document, 23 pp. October 9.
- 12.3. USEPA. 1999. Compendium Method IO-1.1. Determination of PM₁₀ in Ambient Air using the Andersen Continuous Beta Attenuation Monitor. Document No. EPA/625/R-96/010a. June 1999. Available at www.epa.gov/ttn/amtic/files/ambient/inorganic/mthd-1-1.pdf. Accessed 7/20/2006.
- 12.4. Thermo Andersen. 2002. FH62C14- Continuous Ambient Particulate Monitor catalog. Version 18 April 2002.
- 12.5. Pacwill Environmental. 2003. FH62I-N Beta Attenuation Monitor. Available at <http://www.pacwill.ca/betagaug.html>. Accessed on 7/20/2006. Page last updated on October 27, 2003.
- 12.6. SOP B1. 2006. Data Acquisition and Control Hardware. Standard Operating Procedure B1. Purdue Ag Air Quality Lab.
- 12.7. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 12.8. SOP U1. 2006. On-farm Instrument Shelters for Barn Sources. Standard Operating Procedure U1. Purdue Ag Air Quality Lab.
- 12.9. SOP U6. 2006. The Powerware 9125 Uninterruptible Power Supply. Standard Operating Procedure U6. Purdue Ag Air Quality Lab.

13. Contact Information

- 13.1. Manufacturer: Thermo Electron Corporation, 26 Tech Valley Drive, East Greenbush, NY 12061.
 - 13.1.1. Technical Support: 1- 866-282-0430 or 518-452-0065.
- 13.2. PAAQL: odor@purdue.edu, heber@purdue.edu, <http://www.AgAirQuality.com>.

Appendix A

Thermo C-14 Beta Sampler Calibration Form
For Samplers Using Firmware V2.049

Site: _____ Monitor S/N: _____ Date: _____

Temperature Sensor Calibration

Article I. Allow unit to run, calibrate sensors at normal system operating temperatures.
Article II. Calibration Menu – unlock code 147
Article III. Sensors submenu – go through each item in turn and adjust as needed. Log the final results after adjustment.

T₁ ambient sensor _____ Reference Ambient Temp _____ Criteria: ± 6 °C
T₂ upper head sens. _____ Reference upper head _____
T₃ lower head sens. _____ Reference lower head _____
T₄ heated tube sens. _____ Reference heated tube _____

Pressure Sensor Calibration

P₃ ambient _____ Ref. Barometer _____ Criteria: ± 10 mmHg

Note: hPa = mm.Hg * 1.33 , mm.Hg = hPa * .75218 (@20° C)

Flow Rate Calibration

Flow Device Type _____ S/N _____

Sampler Flow _____ Actual Flow _____ Criteria: ± 2%

Note: L/m = L/hr / 60

High Voltage Automatic Set Process

2. Service Menu – unlock keypad - Mechanical Control – turn pump off
3. Perform Filter Change & Zero (FC+Z)
4. Watch for QLF figure to have “**” next to it
5. Calibration Menu – unlock code 147 – High Voltage & Thresholds – Start – allow 5 minutes until stable, log readings in chart provided here
6. Back up on screens and Save to EEPROM
7. Service Menu – Mech. Ctrl – pump on, zero tape, back up & lock keypad, exit menu.

New HV

S_{ref}

Plateau Slope

Mass Foil Calibration

- 1) Operations Menu – unlock keypad if locked – Temp Ctrl System screen – set heater control to Off
- 2) Let sampler run and wait for T₂ = T₁ although being within 2°C seems to be sufficient
- 3) Service Menu – Mechanical Control screen – turn pump off.
- 4) Calibration Menu – Foil Calibration – follow screen prompts step by step
- 5) Note the Amp Values and log them in the chart to the right
- 6) Remove span foil and holder per prompt
- 7) Back up and Save to EEPROM
- 8) Operations Menu, Temp Ctrl – set heater control to On.
- 9) Service Menu, Mechanical Control – ensure that pump is On
- 10) Lock keypad

Foil Span Value μg^*	Old Amp Value	New Amp Value	Bias % of FS
---------------------------------	---------------	---------------	--------------

* For reference only – from the span foil in the calibration kit used for this test.

Values Archived to EEPROM? _____

Comments:

Operator: _____

Date: _____

QA Coordinator: _____

Date: _____

**Appendix B. Monthly Maintenance Checks – Thermo Electron FH 62C14 (Beta)
Continuous Ambient Particulate Monitor**

Site _____ S/N _____ MM/YYYY _____

Date		Clean PM ₁₀	Check Filter Tape	Clean inlet

Flow, Temp, and Press Checks

Date	Parameter (Flow, Temp, or Press)	Ref. Device Value	Beta C-14 Value	Diff.

Criteria : Flow check ± _____% Conversions: Pressure: mmHg = hPa x 0.75218
 (From QAPP) Temp check ± _____°C hPa = mmHg x 1.33
 Press check ± _____ mmHg Flow: L/m = L/h / 60

Comments: _____

**USE OF CONTROL CHARTS FOR PERFORMANCE MONITORING OF
GAS ANALYZERS AND ANALYTICAL INSTRUMENTS**

Standard Operating Procedure (SOP) Q1

**USE OF CONTROL CHARTS FOR PERFORMANCE MONITORING OF GAS
ANALYZERS AND ANALYTICAL INSTRUMENTS
Standard Operating Procedure (SOP) Q1**

Prepared by

Bill W. Bogan and Claude A. Diehl

Reviewed by

Albert J. Heber

Effective Date: November 6, 2006

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions	3
4. Cautions	3
5. Personnel Qualifications.....	4
6. Equipment and Supplies	4
7. Procedures	4
8. Data and Records Management	6
9. Quality Control and Quality Assurance	6
10. References.....	6

1. Scope and Applicability

- 1.1. Control charts provide a real-time graphical representation of the performance of a gas analyzer or other analytical instrument, which can be used to assess data quality and to spot long-term trends in the instrument's performance. The purposes of control charts are as follows:
 - 1.1.1. Visual presentation of the quality control of the analyzers and other instruments
 - 1.1.2. Real-time assessment of the data quality of the analyzers or measurement devices
 - 1.1.3. To monitor the long-term performance of the instrument
 - 1.1.4. To determine when a corrective action must be done
- 1.2. The control charts described in this SOP can be applied to gas analyzers, and to any other type of measurement device (e.g. temperature, relative humidity, and pressure sensors).
- 1.3. A control chart plotting the standard deviation is beyond the scope of this SOP.

2. Summary of Method

Control Charts are generated by plotting data from the zero-precision checks (SOP G9) with known reference standards (control sample). After the zero-precision check is conducted, the control chart must immediately update to give real-time assessment of the instrument's performance. A control sample is used to check the instrument's performance. The instrument's response to a control sample is an indication of the instrument's deviation from the true value that is a result of intrinsic instrumental measurement error. Control limits are established to maintain statistical control of the data quality. If the reference standard results in a measurement outside the control limits, the control chart is a tool used to alert the analyst / researcher to increased uncertainty in the instrument's measurements. Other Control Chart parameters include the mean and standard deviation of the last n samples, where n may be from 5 to 20.

3. Definitions

AirDAC	Air data acquisition and control program
Control Limits	The bounds of the range (above and below the true value) that denote acceptable performance of the analyzer or sensor.
Control Sample	A known reference standard
PAAQL	Purdue Agricultural Air Quality Laboratory
QAPP	Quality Assurance Project Plan

4. Cautions

- 4.1. Use the correct range for the analyzer or sensor, and for AirDAC. A range that is too small will cause the analyzer or sensor's analog output to be pegged in response to the reference standard.
- 4.2. The control sample applied to check the analyzer or sensor must be consistent over time. In the case of gas analyzers, for example, use the same concentration for the reference gas for the zero-precision check (SOP G9).

- 4.3. The control sample should be representative of the range of measurements which the analyzer or sensor is expected to measure. In the case of gas analyzers, verify that the concentration of the reference gas is similar to that in the gas samples being analyzed.

5. Personnel Qualifications

- 5.1. Personnel must be trained in the use of control charts before working with any analyzer or sensor for which a control chart is used.
- 5.2. Each analyst must also read and understand this SOP before conducting a multipoint calibration (SOP G8) and zero-precision check (SOP G9).

6. Equipment and Supplies

- 6.1. Computer and AirDAC software program (SOP B2)
- 6.2. Microsoft Excel software

7. Procedures

- 7.1. Setup of the Control Chart
 - 7.1.1. A template of the Control Chart will be supplied by PAAQL to each site, and will be located in the Instrument Performance Record spreadsheet of each individual instrument. Each instrument (analyzer or sensor) will have its own Control Chart.
 - 7.1.2. General formatting and style (Fig. 1)
 - 7.1.2.1. Use Microsoft Excel's x-y plot function for all Control Charts.
 - 7.1.2.2. Use SI units.
 - 7.1.2.3. The ratio of the width to height of the chart should be 1.62. This ratio is known as the Golden Ratio, and is related to the Golden Rectangle. A template is in the Instrument Performance Record File.
 - 7.1.2.4. Use Arial font in graphs and do not bold.
 - 7.1.2.5. Use 14-point font for the title.
 - 7.1.2.6. Use 12-point font for all other notations (axes, labels, comments, and numbering).
 - 7.1.2.7. Place the tic marks on the outside of the graph.
 - 7.1.2.8. Format each tic label with fixed decimal places.
 - 7.1.2.9. The x-axis is time (Date). Include one tic mark per week (every 7 d), and label every so many weeks with mm/dd such that the labels fit on the axis.
 - 7.1.2.10. Don't use vertical labeling on the x-axis.
 - 7.1.2.11. Put the legend of the graph on the inside of the chart box, not outside.
 - 7.1.2.12. Place captions outside the chart.
 - 7.1.2.13. Avoid redundancies in graph labeling. For example, there is no need for a title, since a caption covers it.
 - 7.1.2.14. Label the control limits.
 - 7.1.2.15. Multiple baselines can be used for one instrument.
 - 7.1.2.15.1. Simultaneous baselines for the precision check gas standard and the zero reference gas can be plotted on the same chart.

7.1.2.16. Ancillary data can be plotted on the control chart (e.g. reaction chamber pressure for a chemiluminescence ammonia analyzer).

7.1.2.17. Include the serial number of the instrument in the control chart.

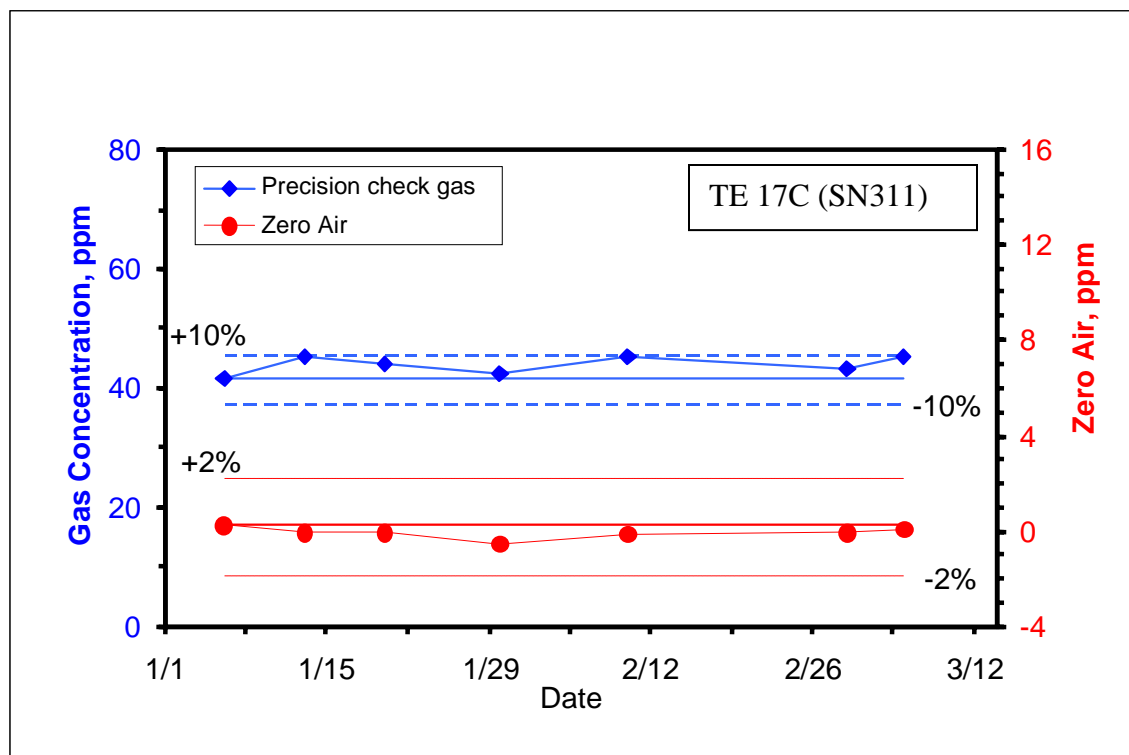


Figure 1. An example of a Control Chart, in this case for a TE 17C Ammonia Analyzer. Two baselines (both components of the zero/precision check) are included, along with their control limits ($\pm 2\%$ for the zero gas and $\pm 10\%$ for the precision check gas).

7.2. Updating the control chart

- 7.2.1. Before updating, save the current control chart in the Instrument Performance Record File's "Zero – Precision Record" worksheet to keep a record of past control charts.
- 7.2.2. The control chart is updated from the zero-precision check data that is tabulated in the Instrument Performance Record File's "Zero – Precision Check" worksheet. This data is entered as described in SOP G9.
- 7.2.3. The baseline measurement is the first zero-precision check after a multipoint calibration (SOP G8) that is the target value.
 - 7.2.3.1. The baseline measurement is established using a standard reference, the value of which is chosen to be consistent with values that are expected to be measured.
- 7.2.4. Save the updated control chart in the Instrument Performance Record File's "Zero – Precision Check" worksheet, as described in SOP G9.

7.2.5. Control limits

- 7.2.5.1. Control limits are used to determine whether the analyzer or sensor has drifted out of calibration.
 - 7.2.5.1.1. Do the following when data points fall outside the control limits:
 - 7.2.5.1.1.1. Access the analyzer's or sensor's operation to determine if the equipment is operating properly or is in need of repair.
 - 7.2.5.1.1.2. Recalibrate the analyzer or sensor, and establish a calibration curve and new control chart
- 7.2.5.2. The controls limits are set to a \pm percentage of the baseline measurement. This percentage is dependent upon the Data Quality Objectives (DQO's) in the QAPP for the gas analyzer or sensor that the Control Chart is monitoring.
- 7.2.5.3. The control limits can be set in the Instrument Performance Record File's "Zero – Precision Check" worksheet. The percentage is entered into the boxes labeled "Control Limit %" for both the precision control limit and the zero control limit.

8. Data and Records Management

- 8.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
- 8.2. Manage all data according to SOP B5.
- 8.3. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes. Overstrike all errors in writing with a single line. Initial and date all corrections.

9. Quality Control and Quality Assurance

- 9.1. Maintain a control chart for all gas analyzers or sensors specified in the QAPP for the project in question.
- 9.2. Update the Control Chart immediately after performing a zero/precision check of a gas analyzer (SOP G9) or other sensor.
- 9.3. Review the Control Chart to assess data quality and instrument performance every time it is updated.
- 9.4. E-mail the Control Charts to the research leaders (PI, QC Manager, Gas Analyzer Manager, etc.) in a timely manner. The exact list of persons who should receive Control Charts, and the frequency, will be specified in the QAPP for the particular project.
- 9.5. Keep a printout of the most recent Control Chart for each analyzer or sensor in the on-farm instrument shelter (OFIS). Keep these copies available for site audits, either by PAAQL personnel or outside QA auditors.

10. References

- 10.1. Taylor, J.K. 1987. Quality Assurance of Chemical Measurements. CRC Press LLC, Boca Raton, FL.

- 10.2. Ingersoll, W.S. 2001. Environmental Analytical Measurement Uncertainty Estimation – Nested Hierarchical Approach. Defense Technical Information Center # ADA396946. Online at <http://www.stormingmedia.us/00/0037/A003793.html>. Accessed 6/23/2006.
- 10.3. SOP B2. 2006. Data Acquisition and Control Software (AirDAC). Standard Operating Procedure B2. Purdue Ag Air Quality Lab.
- 10.4. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 10.5. SOP G1. 2006. The PAAQL Gas Sampling System. Standard Operating Procedure G1. Purdue Ag Air Quality Lab.
- 10.6. SOP G3. 2006. Measurement of Carbon Dioxide (CO₂) with the MSA Model 3600 Infrared Gas Monitor. Standard Operating Procedure G3. Purdue Ag Air Quality Lab.
- 10.7. SOP G4. 2006. Measurement of Ammonia (NH₃) Using the TEI Model 17C Chemiluminescence Analyzer. Standard Operating Procedure G4. Purdue Ag Air Quality Lab.
- 10.8. SOP G5. 2006. Measurement of Hydrogen Sulfide (H₂S) with the Thermo Electron Corporation Model 45C Pulsed-Fluorescence Analyzer. Standard Operating Procedure G5. Purdue Ag Air Quality Lab.
- 10.9. SOP G6. 2006. Measurement of Methane and Non-Methane Hydrocarbons with the Thermo Electron Corporation Model 55C Analyzer. Standard Operating Procedure G6. Purdue Ag Air Quality Lab.
- 10.10. SOP G7. 2006. Use of the INNOVA 1412 Photoacoustic Multi-Gas Monitor. Standard Operating Procedure G7. Purdue Ag Air Quality Lab.
- 10.11. SOP G9. 2006. Zero and Precision Checks of Gas Analyzers. Standard Operating Procedure G9. Purdue Ag Air Quality Lab.
- 10.12. SOP G10. 2006. Measurement of Ammonia Using the MSA Photoacoustic Infrared (IR) Analyzer. Standard Operating Procedure G10. Purdue Ag Air Quality Lab.
- 10.13. SOP G11. 2006. Operation of the Environics[®] Computerized Gas Dilution System. Standard Operating Procedure G11. Purdue Ag Air Quality Lab.

PRODUCER COLLABORATIONS AT BARN MONITORING SITES

Standard Operating Procedure (SOP) S1

PRODUCER COLLABORATIONS AT BARN MONITORING SITES

Standard Operating Procedure (SOP) S1

Prepared by

Albert J. Heber and Bill W. Bogan

Reviewed by

Juan Carlos Ramirez and Lingying Zhao (Ohio State Univ.)

Effective Date: November 6, 2006

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions	3
4. Health and Safety	3
5. Cautions	4
6. Interferences	5
7. Personnel Qualifications and Training	5
8. Utilities, Equipment and Supplies Provided at Farm.....	5
9. Procedures	6
10. Data and Records Management	7
11. Quality Control and Quality Assurance	7
12. References.....	8
Appendix A. Barn Site Information Forms.....	9
Appendix B. Producer Event Form.....	11

1. Scope and Applicability

The success of on-farm air-quality studies depends on communication and collaboration between the research staff, who are vitally interested in collecting high-quality air emission data from the site, and the livestock producer, whose main interest is to operate his/her facility for profit. The livestock producer and his/her staff may or may not have an intense interest in the data. This SOP, therefore, establishes procedures to optimize collaboration between the researcher and the producer, describes research-related information that must be collected from producers, delineates certain expectations, and outlines possible modifications to farm operations that may be needed to accommodate the study.

2. Summary of Method

The facility design and operational information of the farm must be provided by the producer to the researcher. In addition, the researcher must communicate to the producer those requirements which he/she must meet in order to conduct a successful study. The research team must be guided in effective communication with a livestock production entity. The collaboration and communication will include email messages, phone calls, face-to-face meetings, reports, data forms, presentations, data monitoring, and project documentation such as the Quality Assurance Project Plan.

3. Definitions

- | | |
|------------------|---|
| 3.1. SOP | Standard Operating Procedure |
| 3.2. Biosecurity | Preventing spread of disease to or from livestock or poultry. |
| 3.3. QAPP | Quality Assurance Project Plan |

4. Health and Safety

- 4.1. Beware of and do not interrupt worker traffic. Research personnel may request a slight delay or interruption in normal activities during set up and disassembly of instrumentation, but must always defer to the decisions and activities of the farm staff.
- 4.2. Watch out for vehicular traffic such as litter, feed, animal, milk, and egg trucks, loaders, cars, and other vehicles.
- 4.3. The farm's licensed electrician must conduct or approve all electrical work associated with the study. An example is connecting power to the instrument shelter.
- 4.4. Be careful around animals and birds to prevent personal injury.
- 4.5. Beware of moving apparatus such as manure scrapers, manure belts, feed delivery augers and belts, ventilation fans, etc. When working in close proximity to moving equipment, belts, or fans, avoid loose-fitting clothing or dangling jewelry which could become caught in the equipment. Put up or secure long hair.
- 4.6. Ensure that power is disconnected to fans when installing ventilation measurement equipment.
- 4.7. Stay out of confined spaces such as covered manure pits to avoid exposure to potentially dangerous levels of gases. Stay out of the barn when liquid manure in underfloor pits is agitated because manure agitation releases dangerous levels of hydrogen sulfide.

- 4.8. Wear protective clothing to prevent exposure to manure, dust particles, and noxious and toxic gases. Ammonia typically rises above the 35-ppm OSHA short-term exposure threshold.
 - 4.8.1. Research personnel should wear clean gloves when handling the the Producer Event Forms, or any other paperwork, to avoid exposure to potential contamination transferred by farm personnel who may have filled out the form in the barn with contaminated hands.
- 4.9. Emergency evacuation procedures at each measurement location will be those established by the livestock producer.
- 4.10. Post the phone number for medical emergency personnel and directions (map) to the nearest hospital in the instrument shelter close to the entrance.
- 4.11. Maintain a first aid kit in the instrument shelter.

5. Cautions

- 5.1. Notify the producer before any “preliminary” visits (for example, when visiting a site to assess its suitability), or when beginning site setup. Once research has been “established” at the site, it will most likely not be necessary to provide notification of routine weekly site visits, etc. Nonetheless, ascertain and follow the individual producer’s policy in this regard.
- 5.2. Take steps to overcome language barriers (e.g. Spanish vs. English). For example, if the majority of the workers are of Hispanic origin, distribute a pamphlet in Spanish indicating the objectives of the study and the importance of the taking care of the instrumentation. The pamphlet can be complemented with a video in Spanish (possible a PowerPoint presentation with audio in Spanish, or any language needed) that can be distributed to all the research sites, and can be included in a periodic (quarterly) refreshing session (considering that the rotation of workers in the farms can be high).
- 5.3. Post signs or labels by sensitive test equipment that indicates it must not be touched unless authorized by study personnel. Make sure that these are also written in the primary language(s) of the farm workers.
- 5.4. Research personnel must follow all biosecurity protocols and guidelines requested by individual producers. At minimum, these can be expected to include:
 - 5.4.1. Use of disposable boot covers and overalls (Section 8.1)
 - 5.4.2. Cleaning research vehicles and equipment entering the farm according to the producer’s specific protocol
 - 5.4.3. Accessing only areas or barns that are directly involved in the study, and avoiding contact with livestock and areas that the farmer has designated as off-limits
 - 5.4.4. Avoiding contact with dead animals
 - 5.4.5. Avoiding driving across feed delivery lanes to reach the pile or lagoon, or when leaving the pile or lagoon
 - 5.4.6. Ensuring that study personnel coming onto the farm have not exceeded producer-specified limits on foreign travel and/or visits to other poultry or livestock farms
- 5.5. To the greatest extent possible, solicit input and suggestions from the producer as to safe locations for equipment, possible hazards which might occur, and appropriate precautions which might best protect equipment and staff.

- 5.6. Remember that the producer's involvement and time commitment to the project is less than that of the research staff. The amount of time required for the livestock producer or his/her staff to execute their commitments to the study is a major factor influencing current and future involvement. Take care not to use more of the producer's and/or staff's time than is absolutely required for the study.

6. Interferences

- 6.1. Farm should operate with typical management, as though there were no study.
 - 6.1.1. Producers should notify researchers if research equipment is interfering with farm operations and needs to be moved or modified. Producers must agree not to move or modify study equipment themselves, unless failure to immediately move the equipment would result in its damage, or harm to persons or animals.
- 6.2. Tours of the study site must not be given without producer permission. Establish a policy about tours prior to set up of the instrumentation on the farm.
- 6.3. Any add-on research projects at the farm must be approved by the producer.

7. Personnel Qualifications and Training

- 7.1. All producer and research personnel involved in the study must read and understand this SOP.
- 7.2. A pre-study training and orientation session will be conducted by the research team and will include time for the producer to explain the farm operation. The training session will be conducted also in the primary language(s) of the farm workers. Translate any supporting documents or media which are included in the presentation (e.g. pamphlets or videos – Section 5.2) into the primary language(s) of the farm workers.

8. Utilities, Equipment and Supplies Provided at Farm

- 8.1. Personal protective clothing/equipment
 - 8.1.1. Levels of protective clothing will likely vary at a given facility, depending upon the season, current ventilation rate of the barn, and other factors.
 - 8.1.2. Protective clothing may be provided by the researchers, or by the producers, depending, for example, on the preferences of the producer. Reach an agreement on this point with each producer at the beginning of the study.
 - 8.1.2.1. For example, if producers wish to supply their own boots as part of their biosecurity program, researchers will abide by this preference.
- 8.2. Electric power (see SOP U1 Barn Instrument Shelter).
- 8.3. Phone line in instrument shelter
- 8.4. Internet service
- 8.5. A garden hose with a nozzle at the instrument shelter
- 8.6. Restroom and shower facilities, if available
- 8.7. All-weather (e.g. gravel) access to the instrument shelter
- 8.8. Mowing of grass, and/or other weed-abatement measures as necessary, in the vicinity of the instrument shelter

9. Procedures

- 9.1. Detailed information about the farm will be collected for each participating site as part of the site selection process. The site selection criteria and the characteristics of the test site and the barns are in Appendix A.
- 9.2. Producers will allow free access to farm by project staff.
- 9.3. Research staff will obtain photos of the site, preferably from several directions, documenting the locations of buildings and (if applicable) lagoons, topography of site, and placement location of the on-farm instruments shelter(s), Fig. 1.
- 9.4. Producers will be made aware of all necessary structural modifications (e.g. access holes into barns for sensor wires and gas sampling lines). See SOP U2 for Installation of Barn Measurements.
 - 9.4.1. Provide a project schedule and QAPP to the producer at least 90 days prior to beginning any data monitoring.
 - 9.4.2. Set up a monthly phone call with farm management to solve problems, provide updates and facilitate collaboration.
 - 9.4.3. During the course of the study, each collaborating producer is requested to provide the university the following information for each barn (Table A2 in Appendix A):
 - 9.4.3.1. Animal inventory, average weight and average age
 - 9.4.3.2. Animal diet (feed composition, including nitrogen and sulfur levels, and feed consumption)
 - 9.4.3.3. Production (quantity and weight) of marketed animals, birds, eggs, milk, etc.
 - 9.4.3.4. Record of manure removals (for indoor manure storage)
 - 9.4.3.5. Record of cleaning operations
 - 9.4.3.6. Record of animal movements in and out of the barn
 - 9.4.3.7. Record of water consumption
 - 9.4.3.8. Advance notification of any alteration in production schedules and methods
- 9.5. Make sure that the producer understands that placement of equipment may in some cases require that some spaces, cages, or enclosures currently occupied by animals or birds be emptied or modified to include protective structures. Work with the producer to ensure that both the equipment and animals are well-protected.
 - 9.5.1. Trailers which are placed in lots accessible to animals will likely need to be enclosed with protective fencing.
 - 9.5.2. Tiers of layer cages may need to be emptied for placement of sensors (e.g. relative humidity sensors), so that the birds neither damage the sensors nor injure themselves.
 - 9.5.3. Equipment placed in areas which may occasionally be accessed by animals (e.g. alleys in swine barns) may need protective enclosures.
 - 9.5.4. Manure walls may in some cases need to be added to protect equipment.
- 9.6. Livestock producer or his/her staff should contact the PM/PI whenever there are any questions or concerns relating to measurement operations, or if there is catastrophic damage to farm operations.
- 9.7. Computer hardware & software
 - 9.7.1. Data from the Producer Event Form (Appendix B) will be entered into an Excel spreadsheet.
 - 9.7.2. PCAnywhere will be used by the research team to remotely access the data acquisition computer.

10. Data and Records Management

- 10.1. Site Identification. Each site will be labeled in a standard format to denote site type (in this case, area component), location and number. For example, the finisher swine barn site in Indiana is “IN3B”. This format is described in more detail in SOP B5. Producers shall not be identified in each site plan.
- 10.2. Keep a bound notebook at each site that will include a log of farm visits, original copies of Producer Event Forms, and all other written documentation pertaining to the site. Write all entries in indelible ink. Overstrike all errors in writing with a single line; initial and date all such corrections.
- 10.3. The producer will maintain a log sheet (Producer Event Form – Appendix B) of non-monitored farm activities that affect emissions. Examples of pertinent events to record on the Producer Event Form include:
 - 10.3.1. Fan failures, maintenance, or cleaning
 - 10.3.2. Barn cleaning
 - 10.3.3. Major water leaks
 - 10.3.4. Power interruptions
 - 10.3.5. Manure removals
 - 10.3.6. Diet changes (composition or feeding rate)
 - 10.3.7. Molting of birds, or any other change in condition or activity which might influence particulate levels

11. Quality Control and Quality Assurance

- 11.1. Each research team member must maintain a log listing all of his/her exposures to animals at other farms. This will be used to document adherence to the farm’s biosecurity protocol, and to provide information to track infections should any occur.
- 11.2. Monthly conference calls ensure communication between researchers and producer.
- 11.3. During each site visit, verify that farm staff are filling out the Producer Event Form, because they do not normally record such data during daily or weekly activities.
- 11.4. Document all decisions and action items on paper in ink during all planning and status meetings so that it can be confirmed that all parties are in agreement.. Keep copies of these in the bound site notebook (Section 10.2).
- 11.5. Be sure producer has a hard copy of this SOP, the relevant Site Monitoring Plan, and the project management chapter of the QAPP (which will include project management, the rationale for the study and project schedule), and access to the full QAPP on the internet.
- 11.6. The training sessions will include a verification of understanding of the basic aspects of the project, importance of the instrumentation and effects of farm operation on the study. Each attending producer or staff member will be directly engaged in a question and answer session, which may be supplemented with a questionnaire.
- 11.7. Producer should designate a responsible staff member (liaison between the farm and the research team) for collecting and transmitting operational information, and also provide the name of his or her substitute when he or she is not available in the plant. This person should attend all the informational meetings.

- 11.8. Scan all producer event forms, and keep them, both in electronic form (Word or Excel format) and in paper form at the site. Summarize the electronic copy on a weekly or monthly basis, and send this summary to the research leader.

12. References

- 12.1. SOP U1. 2005. On-farm Instrument Shelters for Barn Sources. Standard Operating Procedure. Purdue Ag Air Quality Lab.
- 12.2. SOP B5. 2005. Standard Operating Procedure for Data Management for Barns. Purdue Ag Air Quality Lab.



Figure 1. Typical barn monitoring site with instrument shelter between two monitored barns.

Appendix A: Barn Site Information Forms**Table A1. Site Selection Criteria**

Category	Information
Farm name	
Address, phone	
Producer	Willing to provide production information?
	Cooperative and enthusiastic?
	Willing to make some inconsequential changes to system for the test?
	Willing to train herdsmen or flock managers about importance of test?
	Able to provide information about feed and water consumption?
	Willing to test abatement technologies after the NAEMS?
	Describe management style
Convenience	All weather access?
	Cell phone signal?
	Phone line available for instrument shelter?
	Internet available?
	Lodging nearby for research team?
	Electric power available for trailer?
Barn	Generally describe rations (pelleted, fat added, etc.)
	Describe feed delivery system
	Describe waste collection, handling and treatment system
	Rate barn hygiene from 1 to 5 (1 is cleanest)
Surroundings	Describe surrounding landscape
	Describe other pollution sources (for example, other farms or industrial operations) within one mile of site
Farm	Describe all barns on the site (# hd, size, age)
	Describe land application sites
	List any other animal species on site
	Distance from public road? Type of road (gravel or paved)?

Table A2. Characteristics of test site and barns*.

Descriptive parameters.	Barn 1	Barn 2	Barn 3
Livestock type (layers, etc.) Inventory (number of animals)† # barns at site Year(s) of construction Barn type Barn orientation Distance to site from university, miles Age of animals/birds on 1/1/06 Type of animals/birds (genetics) Average mass, lb Animal occupation, d Number of tiers of pens Shower in/out? Barn width, ft Barn length, ft Barn area, ft ² Ridge height, ft Sidewall height, ft Barn spacing, ft Basement depth, ft Manure collection Manure storage in barn, d Number of pit circulation fans Number of room circulation fans External storage Duration of external storage, d Number of air inlets† Inlet type Inlet adjustment method Inlet control basis Controls vendor Number of exhaust fans† # variable speed fans Largest fan dia., in. Smallest fan dia., in. Fan spacing, in, Fan manufacturer # ventilation stages‡ # temperature sensors† Emergency ventilation Artificial heating Summer cooling Internet service type			

*including the manure storage section, †per barn or room, ††one sampling probe located between the barns represents inlet air for both barns.

**RECORDING AND TRACKING ANIMAL INVENTORY AT LIVESTOCK
BARNs**

Standard Operating Procedure (SOP) S2

RECORDING AND TRACKING ANIMAL INVENTORY AT LIVESTOCK BARN

Standard Operating Procedure (SOP) S2

Prepared by

Sam M. Hanni and Bill W. Bogan

Reviewed by

Albert J. Heber

Effective Date: November 6, 2006

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability.....	3
2. Summary of Method.....	3
3. Definitions.....	3
4. Health and Safety.....	3
5. Cautions	3
6. Interferences.....	4
7. Personnel Qualifications	4
8. Equipment and Supplies	4
9. Procedures	4
10. Data and Records Management	7
11. Quality Control and Quality Assurance	8
12. References.....	8

1. Scope and Applicability

- 1.1. The number, age, and size (life cycle stage) of animals housed in a facility are key factors in determining air emission factors for the facility.
- 1.2. This SOP describes a protocol for collecting information on each of these parameters for use in agricultural air quality monitoring and modeling studies.
- 1.3. This SOP spells out in greater detail the data that individual producers are expected to provide (SOP S1) regarding the animal inventory at their facilities.

2. Summary of Method

The barn inventory for cattle and swine will be determined by counting each animal. For caged layer birds, a selected number or percentage of the cages in each house (as specified in the QAPP) will be counted to obtain a per-pen average, which will then be multiplied by the known number of pens in the house. Since boilers are not housed in cages, an estimate of the total inventory will be obtained by counting the number of cages that hold the birds during transportation and multiplying by the average population in those cages (again, determined by averaging the counts in a prespecified number of cages). The total weight for barns housing swine layers, broilers and cattle will be determined by weighing a percentage of the animals in each barn that is being sampled, averaging the weights, and multiplying by the total inventory.

3. Definitions

- 3.1. **Growth Period:** The duration of time that an animal is in a facility
- 3.2. **Closeout:** The end of a growth period, at which time the barn is emptied
- 3.3. **Portable livestock scale:** A certified scale that is compact and easily moved by two individuals. The scale's range should correspond to that of the animals' beginning and ending weights.

4. Health and Safety

- 4.1. Wear a dust mask and ear plugs while weighing animals.
- 4.2. Use proper lifting technique when lifting or moving animals or scales.
- 4.3. Cattle can become frightened if they are approached too closely, and can cause injury to the individual taking the count. See Section 9.3.1 for procedures for counting cattle.

5. Cautions

- 5.1. Take steps to make sure a correct inventory was taken by the producer.
 - 5.1.1. Collect all inventory records before they are removed from the barn or are lost at the end of a growth period.
- 5.2. Animals that are removed from a cage or pen to be counted or weighed should be returned to the same pen.
- 5.3. Do not startle the animals or chase the animals in a pen.

- 5.4. Make sure that all scales have been properly calibrated (Section 11.1).

6. Interferences

- 6.1. Farm workers may need to interrupt counts to conduct operations. Try to schedule counts so they will not conflict with normal farm procedures.
- 6.2. The movement of animals from one location in a pen to another location in the pen could result in an inaccurate count. When manually counting livestock, verify the numbers by repeating the count.
- 6.3. Make sure that scales are level and free of dirt or manure.

7. Personnel Qualifications

- 7.1. All producer and research personnel involved in the study should read and understand this SOP.
- 7.2. A pre-study training and orientation session will be conducted by the research team and will include time for the producer to explain the farm's operation. The training session will also be conducted in the primary language of the farm workers with help of the translated video or PowerPoint presentation.
- 7.3. Individuals should gain experience in handling animals before moving animals without experienced supervision. Persons who do not have experience catching and handling livestock of the particular species at a site must work with, or be supervised by, farm personnel when directly handling the animals.
- 7.4. Each individual involved in the weighing of livestock must read and understand the manufacturer's manual for each scale used at that site. See Section 11.1.

8. Equipment and Supplies

- 8.1. Clipboard
- 8.2. Inventory worksheet
- 8.3. Pencils
- 8.4. Livestock marker
- 8.5. Portable livestock scale
- 8.6. On-farm livestock scale
- 8.7. Livestock tags
- 8.8. Sorting board

9. Procedures

- 9.1. General procedures
 - 9.1.1. Before data collection begins, collaborate with the producer or site manager.
 - 9.1.1.1. Request access to inventory worksheets.
 - 9.1.1.2. Learn how the producer inventories the livestock in each barn.
 - 9.1.1.3. Obtain copies of the inventory worksheet at the beginning of each growth period after a barn has been filled, and at closeout.

- 9.1.1.4. Determine the type(s) of scale used by the farm to weigh livestock. Obtain a copy of the manufacturer's manual for that model of scale (Section 11.1).
- 9.1.1.5. The QAPP will specify how often inventories should be taken in each barn.
- 9.1.2. Weigh all livestock before it enters the barn and after it has left the barn.
- 9.1.3. Before livestock enter the barn, take an average weight of all animals.
 - 9.1.3.1. Weigh trucks carrying animals to or from the barn (both empty and full weights).
 - 9.1.3.1.1. Weigh each truck immediately before it is filled and immediately after it has been filled.
 - 9.1.3.1.2. If possible, the truck weights should be taken on scales that have been calibrated by the Department of Transportation, or other responsible state agency. If the scale is located at the farm, refer to Section 11.1. The scale must be able to hold both the tractor and the trailer.
 - 9.1.3.2. Obtain all weights by retrieving copies of scale sheets from the producer.
 - 9.1.3.3. To obtain the average animal weight, subtract the empty weight of the truck from the full weight, and divide by the total number of animals on the truck
- 9.1.4. Count the number of animals or birds in each barn.
 - 9.1.4.1. Preferably, two people should work together when counting livestock.
 - 9.1.4.2. Individually count each animal in a pen or cage.
 - 9.1.4.3. Confer and reach agreement on the count before moving to the next pen or cage.
 - 9.1.4.4. Record the count of the pen or cage on the inventory worksheet. If it helps, take pictures of the animals.
 - 9.1.4.5. Calculate the barn inventory.
- 9.2. Swine
 - 9.2.1. For finishers or nurseries:
 - 9.2.1.1. Count pigs on a per-pen basis.
 - 9.2.1.1.1. If possible, stand in the middle of the pen so that the entire pen can be seen.
 - 9.2.1.1.2. With producer permission, mark each animal with a livestock marker to ensure that each animal is counted. One person should do all marking; both should count.
 - 9.2.1.1.2.1. If the pen needs to be recounted, use a different color marker.
 - 9.2.1.1.3. Confer with the individual assisting in the counting.
 - 9.2.1.1.3.1. If the two counts are not the same, immediately recount the pen.
 - 9.2.1.1.4. If the count is correct, record the value and move to the next pen.
 - 9.2.1.1.5. After counting the last pen, add up the total number of pigs.
 - 9.2.1.2. To obtain an average weight of the barn, randomly select pig(s) from a pen, tag them with a livestock tag, and obtain individual weights.
 - 9.2.1.2.1. The total number of pigs which must be weighed to determine the barn average will be specified by the QAPP.
 - 9.2.1.2.2. Use tags to ensure that the same pigs are weighed each time.
 - 9.2.1.2.3. Moving from pen to pen, weigh each tagged pig with a portable scale.
 - 9.2.1.2.3.1. Move each selected pig onto the scale using a sorting board.
 - 9.2.1.2.3.2. Once the pig stops moving, and the weight does not fluctuate, the second person should write down the animal's tag number and weight.
 - 9.2.1.2.4. Mark each pig that is weighed with a livestock marker before it is returned to the pen that it came from.

- 9.2.1.2.5. After all tagged pigs in a pen are weighed, move to the next pen.
- 9.2.2. For gestation, breeding, or farrowing barns:
 - 9.2.2.1. Count the total sows and gilts located in each barn that is being sampled.
 - 9.2.2.2. Obtain an average weight for the barn by weighing individual animals.
 - 9.2.2.2.1. The QAPP will specify the percentage of animals to be weighed during the study, as well as the required frequency.
 - 9.2.2.2.2. Moving from crate to crate or pen to pen, weigh each sow or gilt on a portable livestock scale.
 - 9.2.2.2.3. Record the sow or gilt number, so that it can be identified for future weighing periods.
- 9.3. Dairy cattle in corrals or free stalls
 - 9.3.1. Counting procedures
 - 9.3.1.1. Do not mark or approach cattle. Cattle can become frightened if they are approached too closely, and could cause injury.
 - 9.3.1.1.1. Counters lacking experience around cattle should consult the site manager.
 - 9.3.1.2. Enter the pen, keeping a 10- to 15-ft space between yourself and the animal(s).
 - 9.3.1.3. Both counters should walk from one end of a pen to the other, counting each animal as they pass.
 - 9.3.1.4. If the animals become crowded, walk around the group, making sure that every animal can be seen and counted.
 - 9.3.1.5. Upon completing the count, confer with the individual assisting in the counting.
 - 9.3.1.5.1. If the two counts are not the same, immediately recount the pen.
 - 9.3.2. Weighing procedures
 - 9.3.2.1. Randomly weigh dairy cattle located in the barn or corral where the sampling is taking place. This should be done throughout the study.
 - 9.3.2.1.1. The total number of cows that must be weighed to determine the average, as well as the frequency of weighings, will be specified by the QAPP.
 - 9.3.2.1.2. If the scale is large enough, several cattle can be weighed at once and the average weight can be calculated for each animal.
 - 9.3.2.1.3. If the farm does not have a scale, weigh animals that are being moved onto or off of the farm using a truck scale.
 - 9.3.2.1.3.1. Copy and store all truck scale weigh scale sheets according to SOP B5.
 - 9.3.2.2. Calculate animal weights according to Section 9.1.3.3.
- 9.4. Caged layers
 - 9.4.1. Count layers on a per-cage basis.
 - 9.4.1.1. Contact the site manager to ensure that all cages are in use.
 - 9.4.1.2. Randomly select cages throughout the barn.
 - 9.4.1.2.1. The total number of cages that must be counted, as well as the frequency, will be specified in the QAPP.
 - 9.4.1.3. Both counters should look through the cage and visually count each bird.
 - 9.4.1.4. Upon completing the count, confer with the individual assisting in the counting.
 - 9.4.1.5. If the two counts are not the same, immediately recount the cage.
 - 9.4.1.6. Write down the total number of birds in the cage before moving to the next cage.
 - 9.4.1.7. After all of the required cages have been counted, add up the total number of birds that were counted.

- 9.4.1.8. Divide the number of birds counted by the total number of cages counted to obtain average number of birds per cage.
- 9.4.1.9. Multiply the average number of birds per cage by the total number of cages to obtain an estimate of the total number of birds per barn.
- 9.4.2. To obtain an average weight of the barn, weigh individual birds from each cage.
 - 9.4.2.1. Moving from cage to cage, weigh all the birds in the cage on a portable livestock scale.
 - 9.4.2.2. Mark each bird before it is returned to the cage, so birds are not weighed twice.
 - 9.4.2.3. After all the birds in the cage have been weighed, move to the next cage.
 - 9.4.2.4. Record all cage numbers so that they can be identified for future weighings.
- 9.5. Broilers
 - 9.5.1. Count broilers as they enter the barn.
 - 9.5.1.1. Randomly select boxes of new chicks and count each bird in the box.
 - 9.5.1.1.1. The total number of boxes required will be specified by the QAPP.
 - 9.5.1.2. Count the total number of boxes that are unloaded, and multiply by the average number of chicks in the counted boxes to obtain an estimate of the total number of chicks entering the barn.
 - 9.5.1.3. Verify the all counts (# of boxes, # of chicks in each box) with a partner to ensure accuracy.
 - 9.5.1.4. Compare the number with the total count from the hatchery.
 - 9.5.1.4.1. If the two counts disagree by more than 1%, take the hatchery count as the actual count, as it is more likely to be accurate.
 - 9.5.2. Count broilers as they are loaded into cages for shipment to the slaughtering plant.
 - 9.5.2.1. Randomly select cages as they are being loaded, and count the total number of birds entering the cages.
 - 9.5.2.2. Count the total number of cages that are loaded, and multiply by the average number of birds in the counted cages to obtain an estimate of the total number of birds leaving the barn.
 - 9.5.2.3. Verify all counts (# of cages, # of birds in each cage) with a partner to ensure accuracy.
 - 9.5.2.4. Compare this count with the count at the slaughtering plant.
 - 9.5.2.4.1. If the two counts disagree by more than 1%, take the slaughtering plant count as the actual count, as it is more likely to be accurate.
 - 9.5.3. To obtain an average weight of the barn, catch randomly selected birds throughout the barn.
 - 9.5.3.1. Weigh the birds on a portable livestock scale.
 - 9.5.3.2. The total number of birds which must be weighed will be specified in the QAPP.

10. Data and Records Management

- 10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
- 10.2. Manage all data according to SOP B5.

- 10.3. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes.

11. Quality Control and Quality Assurance

- 11.1. Properly calibrate all truck scales and livestock scales before use.
 - 11.1.1. Livestock scales that are provided by the farm will be calibrated according to the manufacturer's specifications for the particular model of the scale.
 - 11.1.1.1. If the farm cannot provide a manual for the scale, contact the manufacturer directly to obtain a manual.
 - 11.1.1.2. Calibration frequency should be that specified in the manual or every three months, whichever is less.
 - 11.1.2. Truck scales
 - 11.1.2.1. If possible, use truck scales that have been calibrated, maintained, and certified by a state governmental agency.
 - 11.1.2.2. If using a truck scale on a farm, follow the same procedure outlined in Section 11.1.1.
- 11.2. To add up the total livestock count, use a calculator or excel spreadsheet.
- 11.3. If possible, have two people making counts, so that the count can be verified before moving to the next pen, box, or cage.

12. References

- 12.1. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 12.2. SOP S1. 2006. Producer Collaborations at Barn Monitoring Sites. Standard Operating Procedure S1. Purdue Ag Air Quality Lab.

ACTIVITY MEASUREMENTS

Standard Operating Procedure (SOP) S3

ACTIVITY MEASUREMENTS
Standard Operating Procedure (SOP) S3

Prepared by
Juan Carlos Ramirez

Reviewed by
Ji-Qin Ni, Albert J. Heber, Bill W. Bogan,
and Matt J. Darr (Ohio State Univ.)

Effective Date: November 6, 2006

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)
Agricultural and Biological Engineering, Purdue University
West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	4
3. Definitions	4
4. Health and Safety	4
5. Cautions	5
6. Interferences	5
7. Personnel Qualifications.....	5
8. Equipment and Supplies	5
9. Procedures	6
10. Data and Records Management	11
11. Quality Control and Quality Assurance	11
12. References.....	11

1. Scope and Applicability

- 1.1. This procedure refers to the equipment and methods to monitor the activity of animals (dairy cows, pigs, and broiler and layer chickens) and farm workers in confined animal feeding operations (CAFOs) using the Visonic SRN 2000 detector (Figure 1a). It is also applicable to project workers in the on-farm instrument shelter.
- 1.2. Activity data allow correlations to be drawn between animal activity and pollution generation patterns, especially those of the particulate matter (PM), ammonia and carbon dioxide. They also allow for tracking and recording the time(s) when maintenance is conducted in certain areas of the barns, and when site visits are made to the instrument shelter. The activity information for animals and workers helps to interpret aerial pollution data, and to improve quality assurance (QA) and quality control (QC).
- 1.3. This method detects activity which occurs within 18 m of the sensor, depending on detector configuration and mounting. With the standard lens, which has three layers and 38 beams, and a 90° monitoring angle, the maximum coverage is 18 m x 18 m (Figure 1b).
- 1.4. One limitation of the detector, when it is used as an activity sensor, is that the intensity of the output signal also depends on the distance of the infrared emission body. A small movement of a body close to the detector can generate an output signal equivalent to a larger movement of a body farther away from the detector.

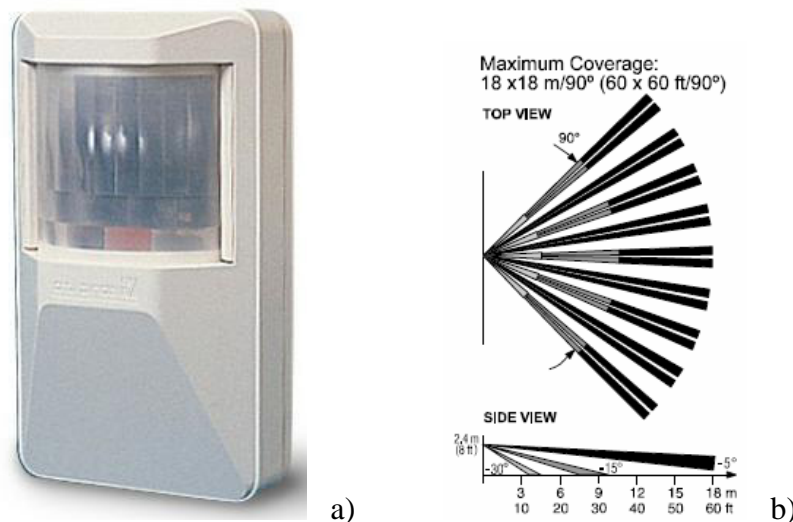


Figure 1. Visonic SRN 2000 detector (a), and its layered beams (b) (Visonic, 2002).

2. Summary of Method

- 2.1. Live animals and workers emit infrared radiation. When animals or workers are active, the infrared sources move. These infrared source movements can be detected by a passive infrared (PIR) detector operating according to the pyroelectric principle.
- 2.2. The pyroelectric sensor inside the PIR detector is made of a crystalline material that generates a surface electric charge when exposed to infrared radiation. The sensor elements are sensitive to radiation over a wide range, so a filter window is needed to establish an incoming range that is most sensitive to animal and human body thermal radiation. The pyroelectric sensor has two sensing elements. A radiation source passing in front of the sensor will activate the first element and then the other. The radiation source must pass across the detector in a horizontal direction if the detector is vertically installed (Glolab, 2006).
- 2.3. This method utilizes the SRN-2000 PIR detector because it provides analog output signals and has a wide detection range. The detector output is proportional to the temperature difference between the object and the background (Pederson and Pederson, 1995), as well as the angular velocity of the object.
- 2.4. The 0-4 VDC sensor output signals are sampled at 1 Hz, converted to 0-2 VDC proportional to motion strength (but inversely proportional to body distance), averaged over 15 s and 1 min, and saved in two data files, respectively, together with other measurement data by the DAQ system.

3. Definitions

- | | | |
|------|--------|--|
| 3.1. | CAFO | Confined Animal Feeding Operation |
| 3.2. | PIR | Passive Infrared Detector |
| 3.3. | LED | Light-Emitting Diode |
| 3.4. | DAQ | Data acquisition |
| 3.5. | AirDAC | Air quality data acquisition and control program |
| 3.6. | OFIS | On-farm instrument shelter |

4. Health and Safety

- 4.1. The sensor will collect dust and fly specks; wear gloves when cleaning and/or maintaining the sensors.
- 4.2. When installing the sensor and lining up the signal cables on the barn ceiling, use the proper ladder height for safe and comfortable installation.
- 4.3. When using an electric drill to install the sensor and its bracket on the barn ceiling or wall, follow safety procedures for routing the electric cable from the electric outlet to the location of the sensor. Install warning signs to alert the farm workers to the location of cable, so as to prevent a tripping hazard.

5. Cautions

- 5.1. When servicing any of the wire connections to the sensor, shut off the exciting power (12 VDC) supply inside the instrument shelter to avoid short-circuits and damage of the sensor and the DAQ system.
- 5.2. Schedule a periodic cleanup of the sensor lens depending on the environment. A dirty lens reduces the sensitivity of the sensor.
- 5.3. When sealing the opening of the sensor for protection, avoid covering the lens.

6. Interferences

- 6.1. There are two small holes (about 3 mm diameter) on the enclosure of the sensor. They may allow dust to enter into the sensor. Place short pieces (about 1 cm long) of electric tape over these holes to seal them.
- 6.2. The orientation of the sensor is of great importance for optimal performance. This orientation needs to be permanently fixed, so that all signals can be compared. Fix the sensor cable so that it will not pull the sensor and change its orientation.
- 6.3. To minimize false activity readings, avoid aiming the sensor at heaters, bright lights, or windows with incident sunlight.
- 6.4. Avoid running the sensor cable close to high voltage electrical power cords.
- 6.5. Avoid running the sensor cable through areas accessible to animals. Animals may chew or otherwise damage cables.
- 6.6. When monitoring animal activity, worker activity will interfere. Locate the sensor to minimize such interferences.
- 6.7. When monitoring worker activity, locate the sensor so that it does not pick up animal activity.

7. Personnel Qualifications

- 7.1. The installer must have some experience with electronic sensors, or have received specific training in the installation of these activity sensors.
- 7.2. The installer and operator(s) must read this SOP and the Visonic Installation Instructions (Visonic, 2002) before installing or operating the instrument.

8. Equipment and Supplies

- 8.1. Passive Infrared Detector (SRN-2000N, Visonic, Ltd., Bloomfield, CT). Vendor Information: ADI, 9745 Industrial Dr, Unit 2, Bridgeview, IL 60455. Phone#: (708) 599-1390, Fax #: (708) 599-1525.
- 8.2. A metallic strip to hold the sensor. This should be approximately 0.5 in. wide and a minimum 7 in. long, flexible enough to be bent by hand, but stiff enough to hold its shape under the weight of the sensor. Such strips are generally available at local hardware stores.

- 8.3. Suitable cable to connect the sensor to the exciting voltage, and to relay signals to the instrument shelter. Use 3-conductor, 22-gage shielded cable for maximum wiring lengths of 230 m, or 20-gage for wiring lengths up to 330 m.
- 8.4. Machine screws to fix the metallic strip to the sensor
- 8.5. Sheet screws or wood screws to fix the metallic strip to the wall or ceiling
- 8.6. Cable ties to secure the cable
- 8.7. Labels to identify the instrument, the objective of the sensor, and a caution to the barn operator(s) to not disturb the sensor. The label also needs to convey the same information in the primary language of the farm workers. It should indicate the name of the farm contact person if any question arises (such as the need to move the sensor(s) because of maintenance or other work).
- 8.8. Electrical tape to close small openings in the sensor body
- 8.9. Phillips or flat screwdriver
- 8.10. Electric or battery operated drill
- 8.11. DC voltmeter (20K ohms/volt) or multimeter to verify the integrity of the sensor if necessary
- 8.12. Protractor or similar instrument to help in positioning the sensor
- 8.13. Sensor power supply (9-16 VDC, 20 mA each sensor) from the OFIS
- 8.14. DAQ hardware and software, and a computer located in the OFIS

9. Procedures

- 9.1. Verify that the lens is correctly selected and has the required coverage and number of beams. Refer to Sections 3.2 “Lens Selection” and 4.4 “Adjusting the Coverage Area” of the Visonic Installation Instructions (Visonic, 2002).
- 9.2. Verify that the lens is properly seated against the sensor body without leaving any visible openings. If there is an opening between the lens and the sensor body, the lens should be re-installed. Refer to Section 3.5 “Change Lenses” and Figs. 7 and 8 of the Visonic Installation Instructions (Visonic, 2002).
- 9.3. Prepare the sensors for field installation. This can include drilling through one of the wiring knockouts (Figure 2) for the cable and one or two mounting knockouts for inserting the screws to fix the sensor to the metallic strip.
- 9.4. Attach the sensor to the metallic strip holder using the machine screws.
- 9.5. Set the Coverage Range Control inside the sensor (Figure 2) to MAX to maximize its detection sensitivity.
- 9.6. Identify and select monitoring locations that allow maximum exposure of target creatures to the sensor’s beams. Monitoring locations may be specified in the project QAPP.
- 9.7. For barn sensors, contact the farm-project liaison to coordinate an installation schedule, and to get authorization to drill at the required locations.
- 9.8. Find electronic outlets for electric tools, if necessary. Ask the farm electrician where to connect the electric tools, if a battery-operated tool set is not available.
- 9.9. Install the sensor cable from the instrument shelter to the monitoring location. Use plastic cable ties to attach the cable to the barn wall or ceiling. Be careful to locate the cable beyond the reach of the animals and away from power lines.

- 9.10. Connect the “T.P.” terminal inside the sensor to the signal conductor that goes to the DAQ analog input channel. Connect the “12 VDC-” and “12 VDC+” to the exciting voltage “-” and “+,” respectively (Figure 2).

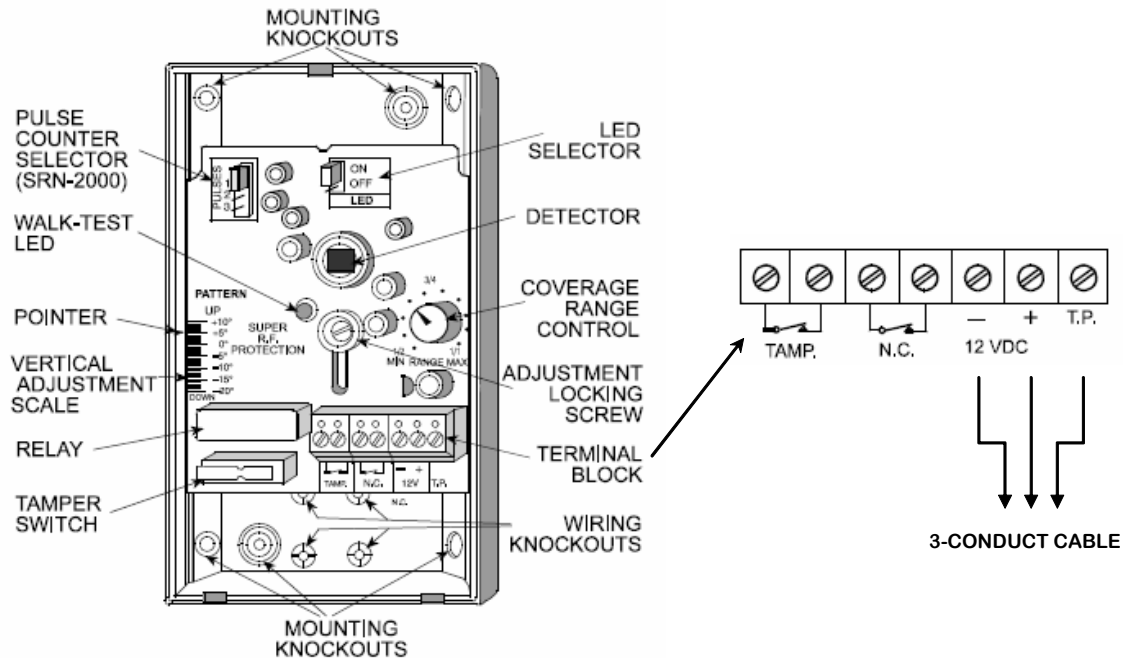


Figure 2. Open-cover view of the sensor and cable connections (adapted from Visonic, 2002).

- 9.11. Verify that the sensor enclosure is tightly closed. Seal the small hole openings to prevent entrance of dust into the sensor using short pieces of electrical tape (Section 6.1).
- 9.12. Fix the metallic holder strip of the sensor to the wall or ceiling. Use two screws so the sensor cannot rotate.
- 9.13. If the sensor is used to monitor animal activities, verify that it is facing the maximum number of animals, yet avoids picking up the activity of workers and/or equipment. See Figure 3 for the installation of the sensor in a layer barn. When installing the sensor in cattle or swine barns, follow the same criteria.
- 9.14. If the sensor is for workers, verify that it covers the maximum area of activities, while avoiding picking up animal activity.
- 9.15. If the sensor is used to monitor activities in the instrument shelter, install it on the end wall to maximize its coverage (Figure 4).
- 9.16. After installation, verify that all connections are correct. Power up sensor by turning on the 12 VDC power supply. Wait for about 5 min to let sensor stabilize.
- 9.17. Test the sensor by moving a hand about 0.5 m in front of it. The “Walk-test” LED (usually a red colored LED) should light up during hand movement. Be sure that the LED selector is set to the ON position (Figure 2). Otherwise, the LED does not light up,

even if the sensor is working properly. Refer to Sections 9.19.1 through 9.19.5 for troubleshooting.

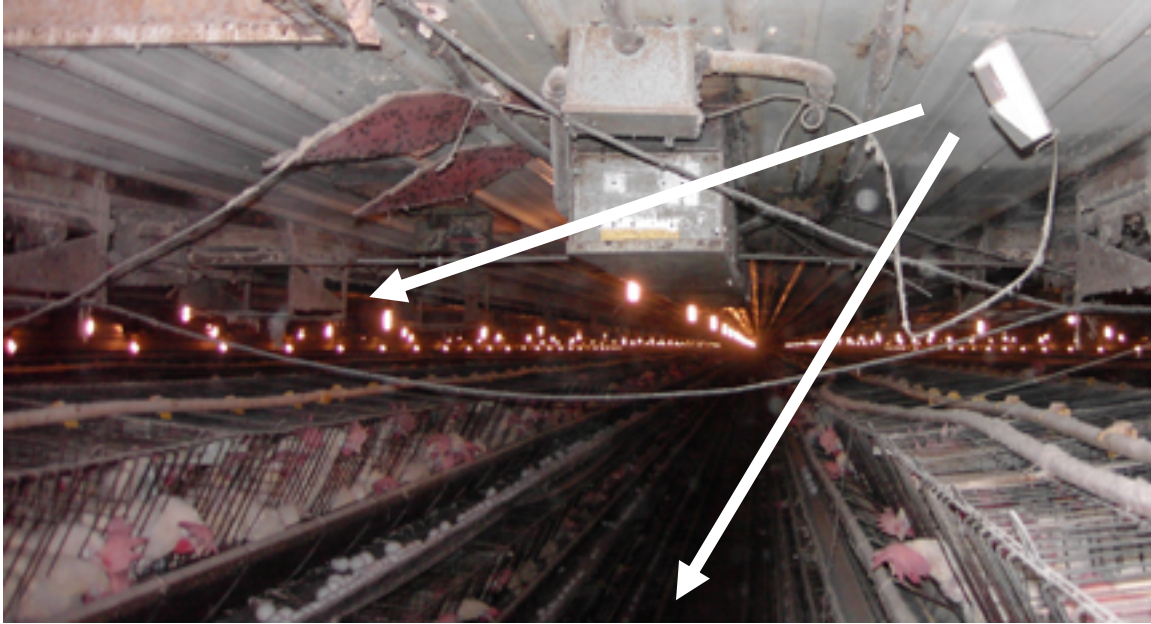


Figure 3. Activity sensor location in an egg layer barn, such that the sensor is aimed at multiple tiers of layer cages.



Figure 4. Activity sensor installed at the end wall inside an instrument shelter.

9.17.1. Verify the signals at the DAQ computer. The history display in AirDAC program (Figure 5. Example activity signals displayed in the AirDAC signal history graphs

(Left: 2-h display. Right: 18-min display). Signals such as those from R5 and R8 are optimal, and represent sensors detecting normal animal activities. Signals such as those from R4 (< 1.0 VDC) indicate that the sensor should be checked. Occasional signals from Door S confirm the presence of workers in the barn.

9.17.2.) should show about 0 VDC when the sensor does not detect activity and between 1 -2 VDC when it does. Refer to Sections 9.19.7 through 9.19.11 for troubleshooting if the signals are abnormal.

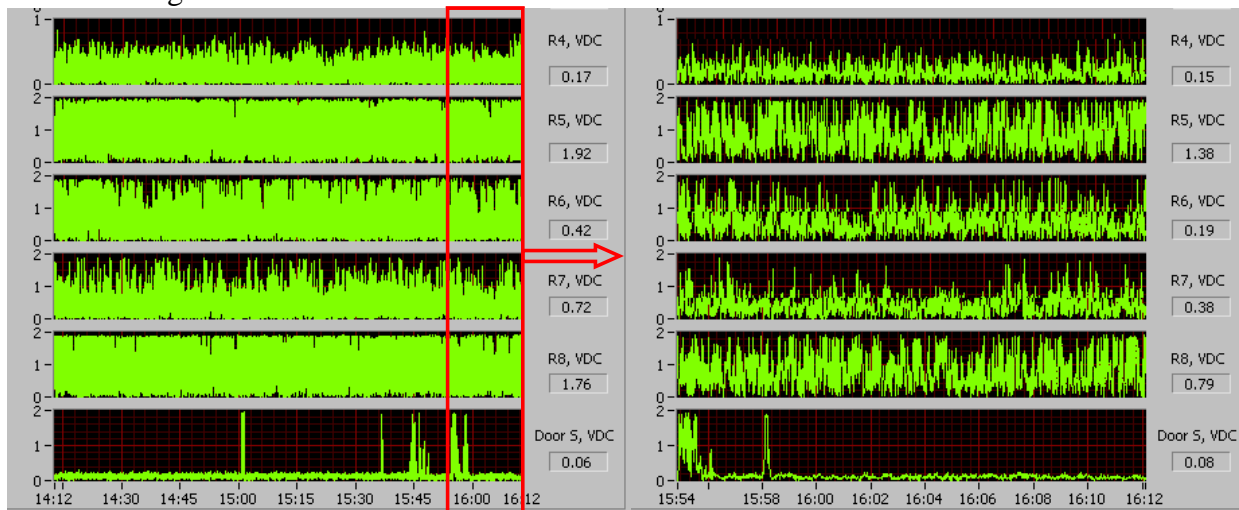


Figure 5. Example activity signals displayed in the AirDAC signal history graphs (Left: 2-h display. Right: 18-min display). Signals such as those from R5 and R8 are optimal, and represent sensors detecting normal animal activities. Signals such as those from R4 (< 1.0 VDC) indicate that the sensor should be checked. Occasional signals from Door S confirm the presence of workers in the barn.

9.18. Instrument calibration

9.18.1. No information is available for sensor calibration or standardization.

9.18.2. A simple method to test the working condition and output signal strength is:

9.18.2.1. To test the background signal when no activity is detected, cover the lens array with a piece of cardboard, so that motion cannot be detected. The output signal should be approximately 2.0 VDC. Refer to Section 4.7 “Test Point” in the Visonic Installation Instructions (Visonic, 2002) for more information. The output signal at the AirDAC should be about 0.0 V.

9.18.2.2. To test an active signal, wave one bare hand about 50 cm in front of the lens. The signals displayed by AirDAC should register the maximum strength of about 2.0 V (Figure 5. Example activity signals displayed in the AirDAC signal history graphs (Left: 2-h display. Right: 18-min display). Signals such as those from R5 and R8 are optimal, and represent sensors detecting normal animal activities. Signals such as those from R4 (< 1.0 VDC) indicate that the sensor should be checked. Occasional signals from Door S confirm the presence of workers in the barn.

9.19. Troubleshooting

- 9.19.1. If the LED still does not light when moving the hand (when the LED selector is set to the ON) there is no sensor signal, open the sensor cover and verify that the exciting voltage is within the required range (9-16 VDC).
- 9.19.2. If no voltage is detected, check the exciting voltage supply at the instrument shelter.
- 9.19.3. If the power supply at the instrument shelter is normal but the sensor does not receive exciting voltage, check cable continuity. Disconnect cable from power supply and DAQ system. Then disconnect it from the sensor terminals. Join all three conductors of the cable together at either the sensor end or the instrument shelter end. Measure the resistance among the cable conductors at the other end using a multimeter to verify continuity.
- 9.19.4. If the cable is abnormal, change it or fix the broken part.
- 9.19.5. If the power supply to the sensor is normal, verify that the sensor lens is clean, outside and inside, and that its orientation is correct.
- 9.19.6. Verify the integrity of the instrument, following the “Test Point” procedure in Section 4.7 of the Installation Instructions (Visonic, 2002).
- 9.19.7. If the sensor signals are not similar to those shown in Figure 5. Example activity signals displayed in the AirDAC signal history graphs (Left: 2-h display. Right: 18-min display). Signals such as those from R5 and R8 are optimal, and represent sensors detecting normal animal activities. Signals such as those from R4 (< 1.0 VDC) indicate that the sensor should be checked. Occasional signals from Door S confirm the presence of workers in the barn.
- 9.19.8. , first verify the DAC page in AirDAC (SOP B2). The DAC page should show about 2 VDC at “15. Sensor sgnl (S)” and about 0 VDC at “17. Adjusted (Y)” for the sensor when it is not detecting activity.
- 9.19.9. The sensor’s setting in the DAC page should be: SL = 2, SH = 3, RL = 0, RH = 1, A = 1, B = 0, and abs = 1.
- 9.19.10. If the sensor displays about 0 VDC in “15. Sensor sgnl (S),” this means AirDAC is not detecting a signal. Measure the voltage of the sensor signal connected to the DAQ hardware terminals using a multimeter. If it is about 2 VDC, the problem is with the DAQ hardware and software. Refer to SOPs B1 and B2 to solve the problem.
- 9.19.11. If the measured sensor signal at the DAQ hardware terminals is about 0 VDC, check the signal wire for continuity.
- 9.19.12. If the wire is normal, check the signal output inside the sensor between terminal 12 VDC – and T.P. (Figure 2). The output signal at the sensor T.P. terminal relative to the ground (-12 VDC) is a voltage, ranging from 0 to 4 VDC. The sensor has an offset of 2 VDC when there is no activity detected. If the sensor signals are more than 2.3 VDC or less than 1.7 VDC when the lens is covered, the unit should be replaced.

9.20. Data acquisition, calculations & data reduction requirements

- 9.20.1. The sensor output signal is acquired with an analog DAQ channel. The signal is sampled every second and calculated in the AirDAC program.
- 9.20.2. The calculated data is averaged every 15 or 60 s, and saved into two separate data files every minute. Refer to SOP B2 for more details.

10. Data and Records Management

- 10.1. Make a record in the test notes after the sensor installation that should at least include: date of installation, location and orientation of the sensor, wiring color code, DAQ channel number to which it is connected, the time the sensor starts collecting signals, and the name(s) of the person(s) who installed the sensor.
- 10.2. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
- 10.3. Manage all data according to SOPB5.
- 10.4. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes. Overstrike all errors in writing with a single line. Initial and date all corrections.

11. Quality Control and Quality Assurance

- 11.1. Verify the sensor orientation periodically, at intervals specified by the QAPP. Document orientation with picture.
- 11.2. Clean the lens surface at a frequency specified by the QAPP.
- 11.3. Correlate the readings from the activity sensor with information provided by the farm liaison and investigate any discrepancy.

12. References

- 12.1. Visonic. 2002. Installation Instructions of Professional All Purpose PIR Detectors SRN 2000 Series. Revision 4 (Jan. 2002), Visonic Inc., Tel Aviv, Israel. Online at [http://www.visonic.com/VisonicHomePage.nsf/sysAllDocuments/64DA845F3663E91EC2256BFD002104E0/\\$FILE/SRN-000%20series%20installation%20instructions.pdf](http://www.visonic.com/VisonicHomePage.nsf/sysAllDocuments/64DA845F3663E91EC2256BFD002104E0/$FILE/SRN-000%20series%20installation%20instructions.pdf). Accessed on 01/02/2006.
- 12.2. Glolab. 2006. How Infrared Motion Detector Components Work. Glolab Corporation, Wappingers Falls, NY. Online at <http://www.glolab.com/pirparts/infrared.html>. Accessed on 02/17/2006.
- 12.3. Pedersen, S. and C.B. Pedersen. 1995. Animal activity measured by infrared detectors. *Journal of Agricultural Engineering Research* 61(4): 239-246.
- 12.4. SOP B2. 2006. Data Acquisition and Control Software (AirDAC). Standard Operating Procedure B2. Purdue Ag Air Quality Lab.
- 12.5. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.

CONTACTOR RELAY MONITORING
Standard Operating Procedure (SOP) S4

CONTACTOR RELAY MONITORING
Standard Operating Procedure (SOP) S4

Prepared by

Lingying Zhao (Ohio State Univ.) and Matthew J. Darr (Ohio State Univ.)

Reviewed by

Ji-Qin Ni, Albert J. Heber and Bill W. Bogan

Effective Date: November 6, 2006

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions	3
4. Health and Safety	3
5. Cautions	4
6. Interferences	4
7. Personnel Qualifications	4
8. Equipment and Supplies	4
9. Procedures	5
10. Data and Records Management	6
11. Quality Control and Quality Assurance	6
12. References	7

1. Scope and Applicability

Process monitoring is critical for analyzing air emission data from livestock facilities. Knowledge of when fans, lights, feeders, manure-removal belts, or other mechanized devices are operating can help interpret changes in air emissions from a barn. This SOP describes the procedures for monitoring production process control at livestock facilities based on the operation of electro-mechanical contactor relays. By utilizing a spare or “dry” contact on the standard contactor relay that controls a device or a group of devices, the data acquisition system can monitor and record the operational status of the devices.

This method usually monitors groups of devices that are collectively controlled by the process control system that the facility uses. For example, if the Stage 2 ventilation for a barn contains five ventilation fans, this method only monitors the on/off signals of the Stage control, and not the individual fans, which can be monitored using the vibration sensor method (SOP A7).

2. Summary of Method

An electrical loop with 5 VDC excitation voltage is created between the data acquisition system and the desired contactor relay. The loop goes through a spare contact in the contactor relay, or an auxiliary relay if no spare contact is available. The loop’s open/closed state is therefore controlled by the spare contact or the auxiliary relay, which depends on the current state of the contactor relay. The loop signals are acquired by the digital input (DI) channels of the data acquisition system every second. The 1-Hz signals are averaged every 15 s and 60 s, and are saved in two separate data files, along with other data acquired from the measurement system (SOPs B1 and B2). The DI signal in the data files are recorded as % of time; for example, if a specific DI channel receives a “closed” signal for 15 s, and an “open” signal for 45 s during one min, the saved datum for this min is “25% of time.”

3. Definitions

- 3.1. DAQ Data acquisition
- 3.2. DI Digital input
- 3.3. OFIS On-farm instrument shelter
- 3.4. QAPP Quality Assurance Project Plan
- 3.5. SMP Site monitoring plan
- 3.6. SOP Standard operating procedure

4. Health and Safety

- 4.1. Only allow certified electricians to connect data acquisition signal cables to the contactor relays.
- 4.2. The signal cable may pass high voltage (120 -240 V) during setup from the facility control system to the OFIS and damage the DAQ system. Do not connect the signal cable to the DAQ system in the OFIS until all setup inside the facility is complete.

5. Cautions

- 5.1. Prevent data acquisition errors caused by corrosion in the monitoring wire by tightly wrapping any exposed portion of wire with electrical tape.
- 5.2. If failures occur from faulty wiring or wiring corrosion, replace the entire loop from the data acquisition system to the process control system.
- 5.3. Coordinate with the farm electrician before working at the electrical control panel to install the signal cable for contactor relay control.
- 5.4. Check the contact terminals before connecting the signal cable. If the terminals are not clean, take out the terminal screws and scratch the dirt off the contacts by a knife or a piece of sand paper to ensure proper wire connection.
- 5.5. Use appropriate size wiring – as specified by the contactor relay manufacturer – to ensure a proper wire/terminal connection.
- 5.6. When running the signal cable(s) from the OFIS to the facility controller, lay the cable(s) properly to avoid tripping hazards to people or animals, and to prevent damage by rodents.

6. Interferences

- 6.1. Manually stopped devices (e.g. a fan is manually unplugged for maintenance) will introduce errors into the monitoring signals. Obtain maintenance records from the facility manager to identify periods when monitored devices may have been shut off, and correct such errors during data processing.
- 6.2. Bad contact connections will cause signal error. Make redundant connections to reduce this type of errors. For example, if there are multiple contactor relays in the same control group, connect two or three contactor relay signals in parallel for this group.

7. Personnel Qualifications

- 7.1. Personnel should be trained in the use of contactor relays and their operation before initiating this procedure.
- 7.2. Personnel working with or near the main process control system must have a sufficient understanding of high-voltage control systems, and the precautions that should be taken in such situations.
- 7.3. Personnel should understand the data acquisition system and read SOPs B1 and B2 for cable connection and setup verification.

8. Equipment and Supplies

- 8.1. Multi-conductor 22-gage (shielded or non-shielded) signal cables for the circuit loop between the data acquisition and the process controller. Refer to Section 9.1.1 for the conductor numbers of the cables.
- 8.2. Data acquisition hardware and software in the OFIS for collecting and recording data, as specified in SOPs B1 and B2
- 8.3. Electrical tape

- 8.4. Cable ties
- 8.5. Multimeter and other electrical tools, e.g. cable cutter
- 8.6. Auxiliary relays if applicable (refer to the SMP in the QAPP for details)

9. Procedures

- 9.1. Prepare for setup
 - 9.1.1. Determine the DAQ signal cable conductor number by referring to the SMP and the design table for DAC measurement and control described in SOP B1. The minimum number of conductors for one barn is the total number of signals (or groups of contactor relays) in that barn plus one for +5 VDC.
 - 9.1.2. Estimate the length of the cables needed to connect the signals from the process controller to the OFIS.
 - 9.1.3. Specify connection requirements, and make a clear wiring diagram, including wire color codes, for the electrician to follow when connecting signal cables to the contactor relays.
 - 9.1.4. Coordinate with the facility manager and the electrician for the cable connection to the contactor relays in the facility.
- 9.2. Run the signal cables
 - 9.2.1. Use a multimeter to check the continuity of the cables to check for defects.
 - 9.2.2. Run the signal cables from the OFIS to near the enclosure(s) of the controller contactor relays. Leave sufficient cable lengths for connecting the contactor relays inside the enclosure by the electrician.
- 9.3. Connecting cables to contactor relays
 - 9.3.1. Identify the spare contacts in contactor relays that can be used for monitoring (Fig. 1).
 - 9.3.1.1. Connect the signal cable wires to the spare contacts in contactor relays according to the wiring diagram.
 - 9.3.2. If spare contactors are unavailable, install auxiliary relays to monitor the system control (Fig. 2).
 - 9.3.2.1. Connect the auxiliary relay actuation coil to the normally open contacts of the contactor relay according to the wiring diagram.
 - 9.3.2.2. Connect the contacts of the auxiliary relays to the signal cables.
- 9.4. Connect the cables to the DAQ system and test DAQ
 - 9.4.1. Use a multimeter to check the signal cable inside the OFIS to verify that no voltage is detected in the cable relative to the ground.
 - 9.4.2. Use a multimeter to check whether a signal wire is closed to the 5 VDC wire when the contactor relay to which they are connected is turned on. The signal wire should be open to the 5 VDC wire when the contactor relay is turned off.
 - 9.4.3. Connect the signal wires of the signal cables to the DI hardware terminals of the data acquisition system according to the wiring diagram and the design table for DAC measurement and control (SOP B1).
 - 9.4.4. Verify the connections to make sure that they are all correct.
 - 9.4.5. Connect the +5 VDC power supply to the exciting voltage (5 VDC) wires of the signal cables.
 - 9.4.6. Power up the DAQ hardware and start the AirDAC software (SOP B2).

- 9.4.7. Verify that the DI channel configurations for these signals are correct in the DAC page of AirDAC. The configuration for these channels should be: Signal low=0, Signal high=1, Range low=0, Range high=1, abs=0, Slope=1, and Intercept=0.
- 9.4.8. Verify that data displayed in the AirDAC and saved in the data files correctly reflect the operational status of the devices.

10. Data and Records Management

- 10.1. Document and maintain the setup records in a workbook of the electronic field notes designated for this method. Supplement this electronic record by a bound record book designated for the method with permanent ink, which should contain copies of the electronic record. Overstrike all errors in writing with a single line; initial and date all such corrections.
- 10.2. Manage all data according to SOP B5.

11. Quality Control and Quality Assurance

- 11.1. Visually inspect data for potential problems according to SOP B3.
 - 11.1.1. Operations of certain devices such as feeders, manure belts, ventilation, and lights are routine. Non-routine behavior should trigger troubleshooting.
 - 11.1.2. Erratic signals (unusual frequency) should trigger troubleshooting.
- 11.2. Verify that signals are correct by observing the process and comparing its status with recorded data.
- 11.3. Verify the fan stage data recorded using this method with the fan monitoring data recorded using vibration sensors (SOP A7). They should agree if both methods are working properly.
- 11.4. For troubleshooting, first study the signals. Specific problems with the DI signals are usually reflected in the data patterns.
 - 11.4.1. If there are no “closed” signals for any DI channel, check the 5 VDC exciting voltage supply. Restore the voltage supply if it is off or disconnected. Check the data acquisition hardware and software to verify that they are working properly.
 - 11.4.2. If there are no “closed” signals for some DI channels connected to a certain cable, check the cable’s continuity. If the cable is disconnected or broken, reconnect it or replace it.
 - 11.4.3. If there are irregular signals for certain DI channels, check the data acquisition hardware for any defect with the specific relevant channels. Replace the defective channel, or replace the DI board.
 - 11.4.4. If the DAQ system is not recording data correctly after the verifications described in Sections 11.2 and 11.3, check the wire connection to the contactor relays for any changes. Also, check with the facility manager for any changes in the production control system or its configuration (e.g. fan stage changes).

12. References

- 12.1. SOP A7. Fan Status Monitoring Using Vibration Sensors. Standard Operating Procedure. Purdue Ag Air Quality Lab.
- 12.2. SOP B1. 2006. Data Acquisition and Control Hardware. Standard Operating Procedure. Purdue Ag Air Quality Lab.
- 12.3. SOP B2. 2006. Data Acquisition and Control Software (AirDAC). Standard Operating Procedure. Purdue Ag Air Quality Lab
- 12.4. SOP B3. 2006. Air Data Pre-Processing software. Purdue Ag Air Quality Lab.
- 12.5. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure. Purdue Ag Air Quality Lab.

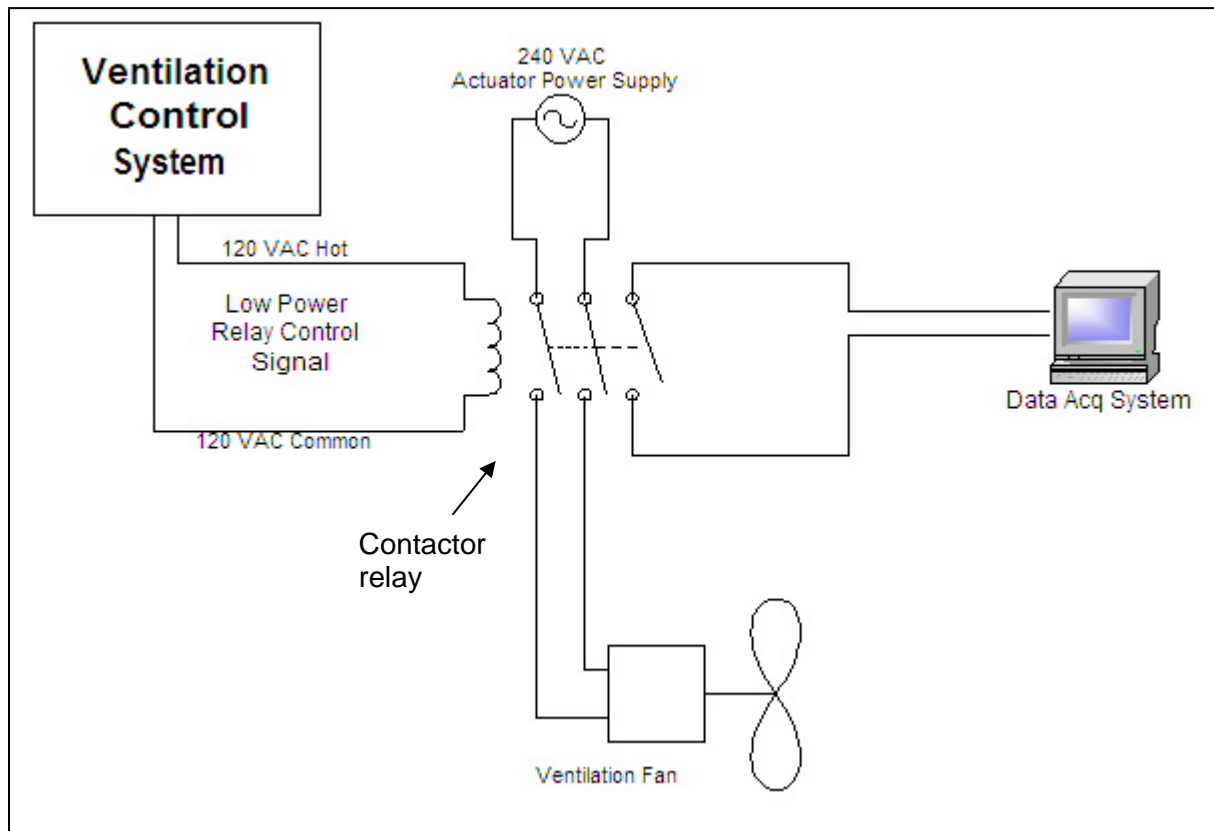


Figure 1. Connection of signal wire to the spare contact in the contactor relay monitor.

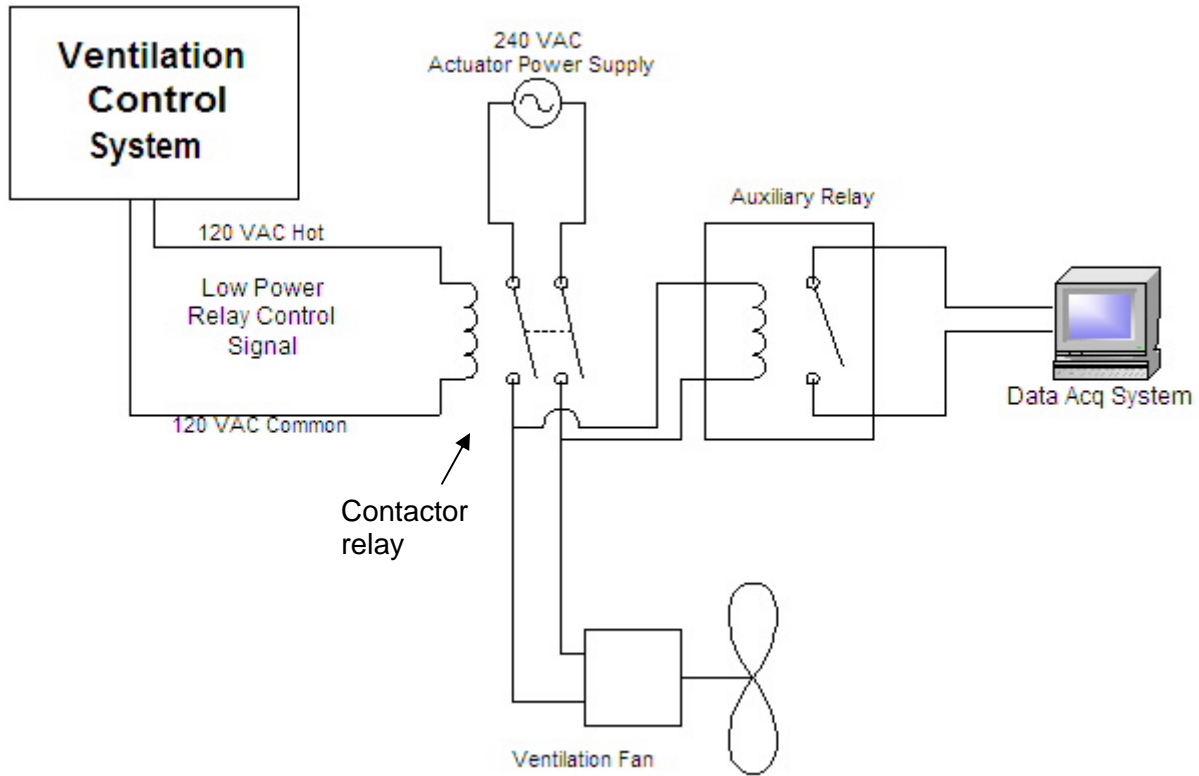


Figure 2. Connection of signal wire to the contactor relay via an auxiliary relay.

DETERMINING NITROGEN BALANCES FROM LIVESTOCK BARNs

Standard Operating Procedure (SOP) S5

DETERMINING NITROGEN BALANCES FROM LIVESTOCK BARNs

Standard Operating Procedure (SOP) S5

Prepared by

Bill W. Bogan and Curt A. Gooch (Cornell Univ.)

Reviewed by

Albert J. Heber

Effective Date: November 6, 2006

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions of Terms and Acronyms	3
4. Interferences	4
5. Personnel Qualifications.....	4
6. Procedures	4
7. Data and Records Management	5
8. Quality Control and Quality Assurance	7
9. References.....	7

1. Scope and Applicability

- 1.1. An ammonia-nitrogen (NH₃-N) balance for a livestock housing facility can provide a check for airborne ammonia emissions that are calculated based on measured air NH₃-N concentrations and the building's air exchange.
- 1.2. An NH₃-N budget makes use of information from several sources including data collected by this project and information available from the literature.
 - 1.2.1. Data for key items such as feed composition, feed consumption, animal inventory, and average animal weight is supplied by the farmers and/or their nutritionists.
 - 1.2.2. Chemical analysis of feed, bedding, manure, milk, and eggs provides additional data.

2. Summary of Method

NH₃-N losses will be estimated using a mass balance approach (Keener et al., 2002). Nitrogen concentrations of all materials, including animal flesh and production (milk and eggs for dairy and egg layers, respectively) entering and leaving the monitored housing facility will be determined or estimated. Feed, fresh bedding, manure (or manure/bedding mixture), milk and eggs (as applicable for each species) will be chemically analyzed for total nitrogen (SOP M4). Literature sources for meat composition for the various species will be used to make assumptions on the N composition of livestock biomass, and biomass gains will be determined based on producer-supplied data (SOP S1) and data from animal inventories conducted by study personnel (SOP S2). Finally, data on feed-consumption quantities and amounts of bedding used will be obtained from the producer (SOP S1). All of these quantities will be determined on a per-unit basis. The QAPP with its site monitoring plans and standard operating procedures will identify sampling locations, times, frequencies, preservation and analysis methods. The sample-collection methods described in SOP M1 will depend on the type of manure handling and storage used at the facility.

3. Definitions of Terms and Acronyms

- | | | |
|------|--------------------|---|
| 3.1. | NH ₃ -N | Ammonia nitrogen |
| 3.2. | QAPP | Quality Assurance Project Plan |
| 3.3. | SOP | Standard operating procedure |
| 3.4. | Total N | Total nitrogen as determined by the Kjeldahl or other method. |
| 3.5. | NF | Nitrogen flux |
| 3.6. | ζ | Animal-specific production efficiency, e.g., eggs per hen per day |

4. Cautions

- 4.1. To develop an accurate model using the equations in Sections 7.3 and 7.4, the manure sample must represent the average holding time encountered by the manure in the monitored housing unit, as well as any manure treatments that occur within the housing unit that may affect the form of N and/or its release to the air. For example, with respect to holding time prior to land application, if only manure that has been stored the maximum time is sampled, its N content will be lower than average, and NH₃ emissions

will be overestimated. Conversely, if the manure sample is fresher than the average storage time, N content will be overestimated, and emissions underestimated. In order to achieve this, it is necessary that the manure be well-mixed prior to or during sampling.

- 4.1.1. If manure is being sampled from inside storage units (either associated with the barn or separate), mix the manure using one of the methods described in SOP M1 prior to collecting the sample.
- 4.1.2. If samples are taken as the manure exits the barn, take similar steps to ensure that a representative sample is obtained.
- 4.2. Some loss of N may occur through routes that are not evaluated under this procedure.
 - 4.2.1. Nitrate losses through surface water runoff or infiltration into soil can occur if manure is outside (and not stored in an appropriate long-term storage) for a period of time (Kohn et al, 1997). However, this may only occur at an outdoor site (such as a dairy corral), and should not be of concern for other dairy sites or other species.
 - 4.2.2. Feed that is stored outside for a period of time may lose some of its N before or when it is being fed to the animal. If feed is stored outside, the N analysis on feed will be conducted as it is provided to the animals, rather than as it is prior to storage.

5. Interferences

- 5.1. Manure transferred from the manure collection systems of another facility will complicate the nitrogen balance estimation.
- 5.2. Any nitrogen entering or leaving the facility (for example, by the routes listed in Section 4.2.1) that cannot be measured or estimated is an interference to the nitrogen balance calculation.

6. Personnel Qualifications

- 6.1. This SOP assumes a familiarity with the use of personal computers, general spreadsheet software (e.g. Microsoft Excel), and/or other data storage and analysis software.

7. Procedures

- 7.1. Data collected under several other SOPs will be used in the calculations which are conducted to determine the nitrogen balance. Because emissions are to be calculated on a per-barn basis, the data for the mass-balance “check” also need to be collected for each individual barn. These data are shown in Table 1.
- 7.2. Calculate the daily N fluxes in inputs (feed) and outputs (eggs, milk) as follows:
 - 7.2.1. Daily nitrogen flux (NF) in feed (kg/animal-day):

$$NF_{D:feed} = R_{Nfeed} * m'_{feed} / n_b$$
 - 7.2.2. Daily N flux in eggs (mg/hen-d):

$$NF_{D:egg} = m_{egg} * \zeta_{egg} * R_{Negg}$$
 - 7.2.3. Daily N flux in milk (mg/cow-d):

$$NF_{D:milk} = \zeta_{milk} * R_{Nmilk}$$

7.3. Manure

7.3.1. The manure storage duration (the total time manure is stored in the animal housing unit) will most likely not coincide with growing period timeframes, nor will it be correlated to milk production. To avoid the errors described in Section 4.1, manure will be sampled at the end of a storage period (in the cases when manure is stored in the housing unit) or when it is about to exit the barn (in the case when no manure storage is present in the barn), in order to accurately estimate the manure and its nitrogen constituents produced by the housed animals. Thus, the other input and output fluxes will be normalized to the in barn manure storage period (Section 7.4).

7.3.2. Determine total nitrogen flux in manure (mg/animal-day) according to:

$$NF_{\text{man}} = R_{N:\text{man}} * W_{\text{man}}$$

Table 1. Data required for a nitrogen balance.

Quantity	Units	Notation	Reference
(All Species)			
Daily feed consumption rate	kg/barn or group	m'_{feed}	SOP S1
Number of animals	animals/barn or group	n_b	SOP S2
TKN content of feed	mg/kg per ration	$R_{N\text{feed}}$	SOP M4
Weight gain (or loss)	kg/animal/growing period	w	SOP S1 & S2
Manure production rate	kg/animal-day	w_{man}	SOP S1
Total N content of manure	mg/kg	$R_{N\text{man}}$	SOP M4
Bedding addition rate	kg/animal-day	w_{bed}	SOP S1
Total N content of bedding	mg/kg	$R_{N\text{bed}}$	SOP M4
(Layers)			
Average egg mass	kg	m_{egg}	SOP S1 & S2
Production efficiency, eggs	eggs/hen-day	ζ_{egg}	SOP S1
Total N content of eggs	mg/kg per egg	$R_{N\text{egg}}$	SOP M4
(Dairy)			
Production efficiency, milk	kg/cow-day	ζ_{milk}	SOP S1
Total N content of milk	mg/kg	$R_{N\text{milk}}$	SOP M4
(Dairy, Swine, Broilers)			
Total N content of biomass	mg/kg	$R_{N\text{biomass}}$	Literature

7.4. Calculate the average per-animal total N flux over the period of time corresponding to the duration of manure storage according to the following equations:

7.4.1. Total N flux in feed:

$$NF_{\text{feed}} = \sum_{D=1}^n NF_{D:\text{feed}}$$

Where n is the total number of number of days in the manure storage period

7.4.2. Total N flux in eggs:

$$NF_{\text{eggs}} = \sum_{D=1}^n NF_{D:\text{eggs}}$$

Where n is the total number of number of days in the manure storage period

7.4.3. Total N flux in milk:

$$NF_{\text{milk}} = \sum_{D=1}^n NF_{D:\text{milk}}$$

Where n is the total number of number of days in the manure storage period.

7.4.4. Total N flux in bedding:

$$NF_{\text{bed}} = \sum_1^n w_{\text{bed}} \times R_{N:\text{bed}}$$

Where n equals the number of bedding additions made during a manure-management period, if that bedding is removed by the manure-management system during that period

7.4.5. Total N flux in animal weight gain:

$$NF_{\text{weight gain}} = R_{N:\text{biomass}} * w$$

Note that it may be necessary to sum biomass gains over more than one growth period.

7.5. Use the following as a check on the N flux determined in Section 7.3.2:

7.6. $NF_{\text{man}} = NF_{\text{feed}} + NF_{\text{bed}} - NF_{\text{egg}} - NF_{\text{milk}} - NF_{\text{weight gain}}$ 7.7. Using the NF_{man} values from Section 7.3.2 (not Section 7.5), calculate NH_3 emissions (EM_{NH_3}) per manure storage period or in barn manure residence time, in units of $kgNH_3/\text{animal}$, as follows, where 1.2143 is used to convert molar mass of N to molar mass of NH_3 :

7.7.1. For layers:

$$EM_{NH_3} = (NF_{\text{feed}} - NF_{\text{egg}} - NF_{\text{man}}) * 1.2143$$

7.7.2. For broilers:

$$EM_{NH_3} = (NF_{\text{feed}} + NF_{\text{bed}} - NF_{\text{egg}} - NF_{\text{weight gain}} - NF_{\text{man}}) * 1.2143$$

7.7.3. For dairy cows:

$$EM_{NH_3} = (NF_{\text{feed}} + NF_{\text{bed}} - NF_{\text{milk}} - NF_{\text{weight gain}} - NF_{\text{man}}) * 1.2143$$

7.7.4. For swine:

$$EM_{NH_3} = (NF_{\text{feed}} + NF_{\text{bed}} - NF_{\text{weight gain}} - NF_{\text{man}}) * 1.2143$$

8. Data and Records Management

- 8.1. Establish a Microsoft Excel spreadsheet which is used specifically for these calculations.
- 8.2. Analytical results for N contents of manure and other materials will be supplied in electronic version by the contract lab(s). Transfer data electronically from these files to the calculation spreadsheet to eliminate errors associated with manual data entry.
- 8.3. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record. Automatically back up electronic records electronically.
- 8.4. Manage all data according to SOP B5.
- 8.5. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes. Overstrike all errors in writing with a single line. Initial and date all corrections.

9. Quality Control and Quality Assurance

- 9.1. Producer should designate a responsible staff member (liaison between the farm and the research team) for collecting and transmitting operational information, and also provide the name of his or her substitute when he or she is not available in the plant. This person must understand the importance of informing research personnel as to any changes in farm operations which might affect the facility's N budget, including (but not limited to) changes in feed or bedding composition, changes in manure handling practices, changes in manure storage times, etc.

10. References

- 10.1. Keener, H.M., D.L. Elwell, and D. Grande. 2002. NH₃ emissions and N-balances for a 1.6 million caged layer facility: Manure belt/composting vs. deep pit operation. Transactions of the ASAE 45:1977-1984.
- 10.2. Kohn, R.A., Z. Dou, J.D. Ferguson, and R.C. Boston. 1997. A sensitivity analysis of nitrogen losses from dairy farms. J. Env Management 50:417-428.
- 10.3. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 10.4. SOP M3. 2006. Determining Solids Content of Manure Samples. Standard Operating Procedure M3. Purdue Ag Air Quality Lab.
- 10.5. SOP M4. 2006. Determining Total (Kjeldahl) Nitrogen Content of Manure Samples. Standard Operating Procedure M4. Purdue Ag Air Quality Lab.
- 10.6. SOP S1. 2006. Producer Collaborations at Barn Monitoring Sites. Standard Operating Procedure S1. Purdue Ag Air Quality Lab.
- 10.7. SOP S2. 2006. Recording and Tracking Animal Inventory at Livestock Barns. Standard Operating Procedure S2. Purdue Ag Air Quality Lab.
- 10.8. SOP S6. 2006. Determining Total Kjeldahl Nitrogen (TKN) Contents of Feed, Bedding, Milk and Eggs. Standard Operating Procedure S6. Purdue Ag Air Quality Lab.

**DETERMINING TOTAL KJELDAHL NITROGEN (TKN) CONTENTS OF
FEED, BEDDING, MILK AND EGGS**

Standard Operating Procedure (SOP) S6

**DETERMINING TOTAL KJELDAHL NITROGEN (TKN) CONTENTS OF FEED,
BEDDING, MILK AND EGGS
Standard Operating Procedure (SOP) S6**

Prepared by

Alan Sutton, Brian Richert, Scott Radcliffe, Dan Kelly and Bill W. Bogan

Reviewed by

Albert J. Heber and Jerome J. King (Midwest Laboratories, Inc)

Effective Date: November 6, 2007

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions	3
4. Health and Safety	4
5. Cautions	4
6. Interferences	4
7. Personnel Qualifications.....	6
8. Equipment and Supplies	6
9. Procedures	7
10. Data and Records Management	10
11. Quality Control and Quality Assurance	10
12. References.....	12

1. Scope and Applicability

- 1.1. Calculating a nitrogen (N) balance for a farm requires a method for determining the N contents of feed, bedding, milk and eggs at a particular barn or facility (Keener et al 2002).
- 1.2. This method determines the total Kjeldahl nitrogen (TKN) content of samples of these materials. Kjeldahl nitrogen is defined as the sum of ammoniacal nitrogen and nitrogen present in organic compounds.
- 1.3. The digestion method described in this SOP is taken from USEPA Method 1687 (2001 draft version), and is therefore also applicable to wastewaters, drinking water or groundwater, and industrial and other wastes.
- 1.4. The distillation and titrimetric procedures are described in USEPA Method 351.3.
 - 1.4.1. This method has a working range of approximately 0.5-10 mg N/gram of sample (wet weight basis).

2. Summary of Method

Feed, bedding, milk and egg samples are collected on the farm for off-site analysis in the laboratory. This method will determine the nitrogen contents of these materials using the Kjeldahl TKN (total Kjeldahl nitrogen) method (SOP M4). Liquid materials (milk and eggs) will be analyzed like slurry manure samples. Dry feed and bedding will be analyzed similar to a dry manure sample. All samples will be subjected to a metal-catalyzed acid digestion to convert the organic nitrogen in the sample to NH_3 . The NH_3 in the digested sample (the sum of nitrogen originally present in the ammoniacal form, plus that which is liberated from organic compounds) is distilled from the rest of the sample, at which point it is captured in a dilute sulfuric acid/boric acid solution which contains a bromocresol green methyl red indicator. The NH_3 concentration of the distillate is then determined by automated titration with sulfuric acid.

3. Definitions

- | | | |
|-------|-------|---|
| 3.1. | COD | Chemical oxygen demand |
| 3.2. | CV | Calibration verification |
| 3.3. | IPR | Initial precision and recovery |
| 3.4. | MDL | Method detection limit |
| 3.5. | MgO | Magnesium oxide |
| 3.6. | MS | Matrix spike |
| 3.7. | NaOH | Sodium hydroxide |
| 3.8. | OPR | Ongoing precision and recovery |
| 3.9. | OSHA | Occupational Safety and Health Administration |
| 3.10. | PR | Precision and recovery |
| 3.11. | QA/QC | Quality assurance/quality control |

- 3.12. Sample batch A group of samples which are analyzed at the same time, up to a maximum of twenty (20). If more than 20 samples are analyzed together, they must be split into more than one batch for the purposes of including QA/QC samples (Section 11).
- 3.13. SOP Standard operating procedure
- 3.14. TKN Total Kjeldahl nitrogen

4. Health and Safety

- 4.1. Use gloves, tongs, and/or suitable sample holders when handling/moving hot samples.
- 4.2. Treat each chemical compound as a potential health hazard, and reduce exposure to chemicals to the lowest possible level.
- 4.2.1. Wear protective clothing (gloves, lab coats, eyewear) when working with chemicals.
- 4.2.2. Conduct all sample digestions in properly ventilated fume hoods. Use a manifold to remove acid fumes.
- 4.2.3. Keep analytical balances and other work areas clean; deal with spills promptly.
- 4.2.4. When using concentrated acids and bases, keep a fully-stocked spill kit (including suitable neutralizers and absorbents) in the work area at all times.
- 4.3. Maintain a current awareness file of OSHA regulations regarding safe handling of the chemicals specified in this method.
- 4.4. Maintain a reference file of material safety data sheets (MSDS), which will be available to all personnel involved in these analyses.
- 4.5. Sulfuric acid digest is classified as a hazardous waste. Dispose of accordingly.

5. Cautions

- 5.1. Considerable heat is generated when dissolving sodium hydroxide (NaOH) in water, or by mixing acids in water. Cool the container in an ice bath when preparing NaOH solutions or mixing acids in water.
- 5.2. Excessive foaming during distillation can cause foam to pass into the condenser, which can contaminate the receiving flask and condenser with alkaline salts. Treat heavily-foaming samples with an anti-foaming agent.

6. Interferences

- 6.1. Keep samples at or below 4°C after collection and during shipping (SOP M1), and prior to analysis, to minimize microbial degradation of sample solids. Analyze all samples within 7 d of collection. Frozen (archived) samples can be maintained indefinitely.
- 6.2. When determining moisture content of wet samples, weigh as quickly as possible after transferring to crucibles, so evaporative water losses do not cause erroneous readings.
- 6.3. Unless extended drying times are required due to crusting of samples, do not exceed the specified drying times. Overdrying can result in negative errors due to volatilization of certain salts and/or organic matter, or heat-induced chemical decompositions, or to positive errors from weight gain through sample oxidation.

- 6.4. If samples form a crust during the drying stage, this can inhibit further evaporation (either of water or volatile solids), and must be dealt with through special handling (either extended drying times or careful physical disruption of the crust).
- 6.5. Following the initial drying step for total solids determination, some samples may retain small amounts of bound water, water of crystallization or interstitial water in crystals. Loss of this water is very slow; these samples may take longer to reach constant weight.
- 6.6. Some samples may exhibit loss of CO₂ as bicarbonate is converted to carbonate. Loss of ammonium carbonate may also occur.
- 6.7. Samples with high levels of oil and grease may require particularly long drying times to reach a constant weight; it may therefore be difficult to obtain incontrovertible results for these samples.
- 6.8. Some dried samples may be highly hygroscopic, containing stronger desiccants than the desiccators themselves. Follow the procedure outlined in Section 5.3 of SOP M3.
- 6.9. Minimize the time that desiccators containing dried samples are open, as samples can adsorb moisture from the air. If strong desiccants are present in the sample (i.e. if weight gain occurs while samples are in the desiccator), use a vacuum desiccator. Do not remove samples from desiccators until immediately prior to weighing.
- 6.10. Use the same balance for all weighing steps involving a particular batch of samples.
- 6.11. Use only NH₃-free water, which can be prepared by passing reagent water through an ion-exchange column that is packed with a mixture of both strongly acidic cation- and strongly basic anion-exchange resins. Regenerate this column periodically according to the manufacturer's instructions.
- 6.12. NH₃ gas in the lab can deposit into the sample. Minimize exposure of samples to air.
- 6.13. Nitrate in large quantities (>10 mg/L) will oxidize NH₃ produced by the digestion and can introduce a substantial negative error. Although this interference cannot be prevented, its effect can be predicted and accounted for on the basis of preliminary nitrate determination of the sample. Whenever high nitrate levels are a potential issue, nitrate analyses of the sample should be conducted first.
- 6.14. Inorganic salts or solids will increase digestion temperature; however, conducting digestion at temperatures in excess of 400°C will cause pyrolytic loss of nitrogen. If high levels of salts and/or solids are suspected, adding extra H₂SO₄ can partially circumvent this problem. Approximately 1 mL H₂SO₄ per g of salt/solid will preserve the proper ratio, and allow digestion to proceed at the target temperature. Monitoring of digestion temperature will indicate if problems are occurring.
- 6.15. Large amounts of organic matter can consume the acid in the digestion reagent, causing digestion temperature to rise above 400°C. Adding 10 mL H₂SO₄ per 3000 mg COD will prevent this. Monitor digestion temperature and pH if this correction is used.
- 6.16. Nitrogen occurring in certain organic compounds will not be measured by this method. These compounds include those in which the nitrogen occurs in the following forms:
 - azide
 - azine
 - azo
 - hydrazone
 - nitrate
 - nitrite
 - nitrile
 - nitro
 - nitroso
 - oxime
 - semicarbazone

6.16.1. In general, the levels of these classes of compounds in manure samples are not expected to be significant.

6.16.2.

7. Personnel Qualifications

- 7.1. Personnel should be trained in the use of the equipment before initiating the procedure.
- 7.2. Each analyst must read and understand this SOP before initiating these procedures, and must show the ability to generate acceptable data accuracy and precision. Each analyst must also have read and understood PAAQL SOPs M3 and M4, as well as USEPA Methods 351.3 and 1687, as the methods described in this SOP are derived closely from these.

8. Equipment and Supplies

- 8.1. Copies of PAAQL SOPs M3 and M4, as well as USEPA Methods 351.3 and 1687
- 8.2. Total solids determination (SOP M3)
 - 8.2.1. Drying oven
 - 8.2.2. Muffle furnace
 - 8.2.3. 15-100-mL crucibles or evaporating dishes made of porcelain or high-silica glass
 - 8.2.4. Watch glasses of suitable size to cover the crucibles/evaporating dishes
 - 8.2.5. Analytical balance: Sartorius Model CP124S (Capacity = 120 g; Accuracy = 0.0001g), Sartorius AG, Goettingen, Germany), or equivalent.
 - 8.2.5.1. Reference masses: 2-mg, 1000-mg, and 50-g class "S" weights
 - 8.2.6. Magnetic stirrer
 - 8.2.7. Spatulas
 - 8.2.8. Desiccator and desiccant (Drierite, Anhydrous Calcium Sulfate)
- 8.3. Total N determination (SOP M4)
 - 8.3.1. Tecator Instrument Model 2020 digester block and aspirator for sample digestion
 - 8.3.2. Foss Kjeltac Model 2300 automated distillation and titration analyzer (FOSS Analytical, Hilleroed, Denmark), or equivalent
 - 8.3.3. Flasks: 100-mL Kjeldahl and 50-mL Erlenmeyer flasks
 - 8.3.4. Reagents
 - 8.3.4.1. Sulfuric acid: Concentrated, 0.020N & 0.25N H₂SO₄, certified (Midland Scientific, Omaha, NE)
 - 8.3.4.2. Sodium hydroxide: 40% NaOH solution, certified (Midland Scientific, Omaha, NE)
 - 8.3.4.3. Digestion reagent: TT-43 Pro Pac Kjeldahl digestion tablets (Alfie packers, Inc, Omaha, NE)
 - 8.3.4.4. Boric acid indicator
 - 8.3.4.4.1. Add 0.10 g bromocresol green, 0.10 g methyl red, 100 g boric acid, and 20 mL of 0.10N NaOH to a 2-L flask that is half-full of NH₃-free water and dissolve all reagents
 - 8.3.4.4.2. Add NH₃-free water to the 2-L mark on the flask.

- 8.3.4.4.3. Quantitatively transfer contents of the flask to the receiving solution reservoir below the Foss Kjelttech Model 2300.
- 8.3.4.4.4. Add 8 more L of NH₃-free water to the reservoir, for a total of 10 L of solution.
- 8.3.5. Standards
 - 8.3.5.1. Nicotinic acid standard (Contains 100 mg/L organic N): Dissolve 21.637 g nicotinic acid (C₆H₅NO₂) in ~ 150 mL NH₃-free water. Dilute to 200 mL with NH₃-free water.
 - 8.3.5.2. Calibration standards
 - 8.3.5.2.1. Prepare calibration standards by spiking 5-g aliquots of blank sand or diatomaceous earth (Section 8.3.6) directly with nicotinic acid standard solution (Section 8.3.5.1).
 - 8.3.5.2.2. Prepare a minimum of five calibration standards, at five different concentrations, to produce a calibration curve with at least five points.
 - 8.3.5.2.3. At least one of the calibration standards should have an analyte concentration at or below that necessary to meet the data quality objectives of the project. This concentration will be defined in the QAPP for the particular project.
 - 8.3.5.2.4. The remaining standards should correspond to the range of concentrations found in actual samples, but should not exceed the working range of the titration-based system.
 - 8.3.5.3. Calibration Verification (CV) standard
 - 8.3.5.3.1. Prepare the CV standard so its N concentration approximates the midpoint of the calibration curve.
 - 8.3.5.3.2. Use a different source (a different bottle of the same compound, or a different compound altogether) to prepare the CV standard than that used to prepare the calibration standards.
 - 8.3.5.3.3. If a different ammonium compound is used for the CV stock, adjust the amount weighed according to the ratio of NH₃-N atomic weight to the molecular weight. See Section 8.9.4.3 of SOP M4 for an example of such a calculation.
 - 8.3.5.4. Quality Control Sample (QCS): Standard Reference Material 1570a (Spinach Leaf). National Institute of Standards & Technology, Gaithersburg, MD.
- 8.3.6. Blank sand and diatomaceous earth
 - 8.3.6.1. Bake 500 g diatomaceous earth or clean sand at 400°C for 8 h. Cool and store in a glass container with a sealing lid.

9. Procedures

9.1. Total and non-volatile solids contents

9.1.1. Preparation

- 9.1.1.1. As only total solids are to be determined, heat crucibles/evaporating dishes and watch glasses to 103°C to 105°C for 1 h.
- 9.1.1.2. Cool and store all glassware in a desiccator.
- 9.1.1.3. Prior to use, weigh empty clean crucible and watch glass, and record value (to the nearest 0.01 g) of the two combined.

9.1.2. Sample handling

9.1.2.1. Obtaining subsamples

9.1.2.1.1. Milk: Pipette a 25- to 50-g aliquot (25 to 50 mL) to a crucible/dish. Replace watch glass and re-weigh; record weight to the nearest 0.01 g.

9.1.2.1.2. Eggs: Collect entire white and yolk, discard shells. Using a magnetic stirrer, Waring blender, or equivalent, stir or blend thoroughly to homogenize. Taking care that the sample remains homogeneous (i.e. that the yolk and white material do not separate), transfer a 25- to 50-g aliquot to a crucible/dish using a pipette, spoon, or other appropriate tool. Do this with the crucible/dish on a balance, to ensure that the amount transferred is in this range. Spread evenly over the crucible. Replace watch glass and re-weigh; record weight to the nearest 0.01 g.

9.1.2.1.3. Feed or bedding: Mix. Transfer a 25- to 50-g aliquot to a crucible/dish using a spatula, spoon, or other appropriate tool. Spread evenly over the crucible. Replace watch glass and re-weigh; record weight to the nearest 0.01 g.

9.1.2.2. Include one blank (SOP M3, Section 11.8) and one Ongoing Precision and Recovery sample (SOP M3, Section 11.3) with each batch of samples (Section 3.12).

9.1.2.3. Put in oven to dry for 12 h at 103°C to 105°C.

9.1.2.4. Remove from oven with tongs and place in the desiccator to cool to room temperature. Do not remove from desiccator until immediately prior to weighing. See Section 5.3 of SOP M3 if working with highly hygroscopic samples.

9.1.2.5. Weigh cooled crucible/watch glass/sample, and record value to the nearest 0.01 g.

9.1.2.6. Heat the sample at 103°C to 105°C for another hour, cool to room temperature in desiccator, and re-weigh. If weight change exceeds 4% or 50 mg (whichever is less), repeat this step until the weight change drops below this threshold.

9.1.2.7. Record final value of the crucible/watch glass/dry sample to the nearest 0.01 g.

9.1.3. Calculations

9.1.3.1. Wet sample weight =

Crucible/watch glass/wet sample (Step 9.1.2.1) – Crucible/watch glass (Step 9.1.1.4)

9.1.3.2. Dry sample weight =

Crucible/watch glass/dry sample (Step 9.1.2.7) – Crucible/watch glass (Step 9.1.1.4)

9.1.3.3. % Total Solids =

$$\left(\frac{\text{Dry sample weight}}{\text{Wet sample weight}} \right) * 100$$

9.2. Nitrogen content

9.2.1. Sample preparation

9.2.1.1. Obtain percent total solids of each sample to be analyzed, according to SOP M3.

9.2.1.2. Label digestion tubes as necessary to accommodate all samples in duplicate, plus

various QA standards (Precision and Recovery Samples, Calibration Verification Standards, Quality Control Standards, Matrix Spikes, and/or Blanks) as required (Sections 11.2.3 - 11.2.8 state how often each of these must be included).

- 9.2.1.3. Solid samples (feed, bedding): Bring sample to room temperature if necessary. Thoroughly grind the sample. Weigh 1-2 g of sample into weighing paper, fold and transfer to a 250-mL digestion tube. Add 1 Kjeldahl Digestion Mixture tablet and 20 mL of concentrated H₂SO₄, and place tube on the Tecator Model 2020 heating block.
- 9.2.1.4. Liquid/slurry samples (milk, eggs): Add 1.1 g of Digestion reagent (Section 8.3.4.3) to a 50-mL digestion tube. Bring sample to room temperature if necessary. For eggs, mix thoroughly (Section 9.1.2.1.2). Pipette sample (0.5-1 g for eggs (Costa et al 1974) or ~ 5 g for milk (Barbano et al 1990)) to the tube. Add 2-3 glass beads, along with 5 ml of concentrated H₂SO₄. Place tube on the Tecator Model 2020 heating block.
- 9.2.2. Digestion
- 9.2.2.1. Immediately set up the exhaust system, and place the exhaust manifold so that it covers the mouth of each digestion tube. Turn water to vacuum on low.
- 9.2.2.2. Digest for 2 h.
- 9.2.2.2.1. Be sure that the manifold and hood fan are operating properly to remove the fumes released in the digestion process.
- 9.2.2.3. Remove tubes from digestion block with tongs and transfer to cooling tray to cool, leaving the exhaust manifold in place for a minimum of 30 min after the end of the digestion. After 30 min, turn off water to vacuum, and remove exhaust manifold.
- 9.2.2.4. Add NH₃-free water to each tube to the level of the top horizontal support on the cooling rack (~50 mL). Place samples in the cooler and cool to <20 °C.
- 9.2.3. Distillation
- 9.2.3.1. Place samples on the Model 2300 and select “Kjeldahl 5”. This selection ensures that the instrument will dispense a sufficient amount of NaOH to the sample.
- 9.2.3.1.1. Use 0.020 N H₂SO₄ for low-N samples, and 0.25 N for all others. Most samples associated with livestock manure, feed, and bedding are assumed to require 0.25 N H₂SO₄.
- 9.2.4. Calculations
- 9.2.4.1. Calculate TKN (in units of mg/g) in the sample as follows, correcting the amount of titrant (0.25N H₂SO₄) added to each sample by subtracting that needed to titrate a blank to endpoint:

$$\text{TKN} = \frac{\text{Titrant}_{\text{sample}} (\text{mL}) - \text{Titrant}_{\text{blank}} (\text{mL})}{\text{Sample wt (g)}} \times 3,500^*$$

*If 0.25N H₂SO₄ is used in the distillation/titration step. If 0.02 N acid is used, replace 3500 with 280.

10. Data and Records Management

- 10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
- 10.2. Manage all data according to SOP B5.
- 10.3. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes. Overstrike errors in writing with a single line. Initial and date all corrections.

11. Quality Control and Quality Assurance

- 11.1. Total solid and ash (non-volatile solids) contents
 - 11.1.1. Quality Control spiking solutions
 - 11.1.1.1. The composition of a suitable QC spiking solution is given in Section 11.1 of SOP M3. A suitable commercially available product is also listed.
 - 11.1.2. Analyst proficiency demonstration
 - 11.1.2.1. Each new analyst must process a set of Initial Precision and Recovery (IPR) samples as described in Section 11.2 of SOP M3, and meet the performance specifications defined therein.
 - 11.1.3. Ongoing Precision and Recovery (OPR) samples
 - 11.1.3.1. Include one OPR sample (25-50 mL) with each sample batch (Section 3.12), and subject it to all steps of the analytical method.
 - 11.1.3.2. Procedures for preparing OPR solutions, and the required performance ranges, are given in Section 11.3 of SOP M3.
 - 11.1.4. Blanks
 - 11.1.4.1. Include one blank (25-50 mL of deionized water) with each sample batch (Section 3.12), and subject it to all steps of the analytical method.
 - 11.1.4.2. If solid material is detected in the blank at or above the MDL, analysis must be stopped until the source of contamination is identified and eliminated (i.e. a new blank shows no evidence of contamination).
- 11.2. TKN determinations
 - 11.2.1. Reagent purity
 - 11.2.1.1. Use only NH₃-free water in this method. See Step 6.9 for the methodology used to remove NH₃ from water for use in these procedures.
 - 11.2.1.2. All other reagents will be ACS Reagent Grade or better.
 - 11.2.2. Initial demonstration of analyst & laboratory proficiency
 - 11.2.2.1. Every new analyst performing the TKN procedures in this SOP must demonstrate, as described in detail in SOP M4, an ability to achieve acceptable results with the method. The analyst must determine a Method Detection Limit (MDL), and demonstrate linear results ($R^2 \geq 0.96$) with the five-point range of calibration standards (Section 8.3.5.2). He/she must also analyze an Initial Precision and Recovery (IPR) sample set, Blank(s), and Calibration Verification (CV) standard(s).

- 11.2.3. Ongoing Precision and Recovery (OPR)
 - 11.2.3.1. MDLs
 - 11.2.3.1.1. The procedure for determining MDLs is given in Section 11.3.1.1 of SOP M4.
 - 11.2.3.1.2. Re-determine/verify MDLs at least once every year.
 - 11.2.3.1.3. Re-determine/verify MDLs if the method or analytical system is modified.
 - 11.2.3.2. Precision and Recovery (PR) samples
 - 11.2.3.2.1. Each batch of 10 samples must contain a minimum of one PR sample.
 - 11.2.3.2.2. Directions for preparing and analyzing the PR samples, as well as the required performance limits, are included in Section 11.4.2 of SOP M4.
 - 11.2.3.2.3. All samples must be associated with an acceptable PR standard before their results may be reported.
- 11.2.4. Calibration curves
 - 11.2.4.1. Generate a new calibration curve (containing a minimum of five points) whenever a new standard is obtained.
- 11.2.5. Calibration Verification (CV) standards
 - 11.2.5.1. Run a Calibration Verification Standard (solid or slurry as appropriate based on the sample type) once per every ten analyses.
 - 11.2.5.1.1. Acceptance criteria for the CV Standard is $100\% \pm 5\%$.
 - 11.2.5.1.2. If the CV does not meet the acceptance criteria, identify and correct the problem, including possible recalibration of the system.
 - 11.2.5.1.3. All samples must be associated with an acceptable CV Standard before their results may be reported.
- 11.2.6. Quality Control standards
 - 11.2.6.1. Analyze a QC standard with each batch of 20 samples run in one day, or every 12 h, whichever is more frequent.
- 11.2.7. Matrix Spikes
 - 11.2.7.1. To assess the performance of the method on a given sample matrix, spike (in duplicate) a minimum of 5% (1 sample in 20) of the samples from a given site.
 - 11.2.7.2. Procedures for determining the required spiking level, assessing spike recovery, and evaluating the acceptability of results are given in SOP M4 (Section 11.8).
- 11.2.8. Blanks
 - 11.2.8.1. Run a minimum of one blank with each batch of 20 samples.
 - 11.2.8.1.1. Use 10 mL of NH_3 -free water as the blank for milk and egg samples, and a 5-g aliquot of blank sand as the blank for feed or bedding samples.
 - 11.2.8.2. If N is detected in the aqueous or solid blank at a concentration greater than the aqueous or solid MDL, halt analysis of samples until the source of contamination is eliminated and a new blank shows no contamination.
 - 11.2.8.3. All samples must be associated with an uncontaminated laboratory blank before their results may be reported.
- 11.3. General
 - 11.3.1. Each time any modification is made to this method, the analyst will verify that the modified method meets or exceeds the Method Detection Limit and Initial Precision and Recovery achieved by the same analyst using the original (i.e. unmodified) method. Procedures for this, and for fully documenting all changes made to the method, are given in SOP M3.

11.3.2. Replication

- 11.3.2.1. Run duplicates for a minimum of 1 in 10 samples (10%), with at least one duplicate per sample batch.
- 11.3.2.2. Replicate samples for solids determination must agree to within 10% of their average (% total solids); if not, rerun these samples.
- 11.3.2.3. Replicate samples for TKN determination must agree to within 10% of their average TKN content; if not, rerun these samples.

11.3.3. QC Charts

- 11.3.3.1. Track all IPR, OPR, Calibration Verification, QC Standard, MS, and Blank results to provide graphical representations of continued laboratory performance.

11.3.4. Instrument Calibration and Standardization

- 11.3.4.1. Calibrate analytical balance annually (at a minimum). See SOP M3 for procedures for calibrating balances.

12. References

- 12.1. USEPA Method 1687. 2001. Total Kjeldahl Nitrogen in Water and Biosolids by Automated Colorimetry with Preliminary Distillation/Digestion (Draft). EPA-821/R-01-004 (CD). USEPA Office of Water, Office of Science and Technology. Washington, DC.
- 12.2. USEPA Method 351.3. 1978. Nitrogen, Kjeldahl (Colorimetric, Titrimetric, Potentiometric). Online at http://www.umass.edu/tei/mwwp/acrobat/epa351_3Norg.pdf. Accessed 8/6/2007.
- 12.3. Hoskins, B. 2003. "Laboratory Procedures". Recommended Methods of Manure Analysis (J. Peters, ed.). University of Wisconsin-Madison Cooperative Extension Publishing, pp. 12-13. Online at <http://learningstore.uwex.edu/Recommended-Methods-of-Manure-Analysis-P106C0.aspx>. Accessed 9/6/2007.
- 12.4. Watson, M., A. Wolf, and N. Wolf. 2003. "Total Nitrogen". Recommended Methods of Manure Analysis (J. Peters, ed.). University of Wisconsin-Madison Cooperative Extension Publishing, pp. 18-24. Online at <http://learningstore.uwex.edu/Recommended-Methods-of-Manure-Analysis-P106C0.aspx>. Accessed 9/6/2007.
- 12.5. Keener, H.M., D.L. Elwell, and D. Grande. 2002. NH₃ emissions and N-balances for a 1.6 million caged layer facility: Manure belt/composting vs. deep pit operation. Transactions of the ASAE 45:1977-1984.
- 12.6. Costa, G., M.E. Kerins, F. Kantor, K. Griffith, and W.B. Cummings. 1974. Conversion of Protein Nitrogen into Gaseous Catabolites by the Chick Embryo. Proceedings of the National Academy of Sciences, USA 71:451-454.
- 12.7. Barbano, D.M., J.L. Clark, C.E. Dunham, and J.R. Fleming. 1990. Kjeldahl Method for Determination of Total Nitrogen Content of Milk: Collaborative Study. Journal of the Association of Official Analytical Chemists 73:849-859.
- 12.8. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 12.9. SOP M1. 2006. Manure Sampling. Standard Operating Procedure M1. Purdue Ag Air Quality Lab.

- 12.10.SOP M3. 2006. Determining Solids Content of Manure Samples. Standard Operating Procedure M3. Purdue Ag Air Quality Lab.
- 12.11.SOP M4. 2006. Determining Total (Kjeldahl) Nitrogen Content of Manure Samples. Standard Operating Procedure M4. Purdue Ag Air Quality Lab.
- 12.12.EN SOP 65e. 2004. Total Kjeldahl Nitrogen (TKN). Standard Operating Procedure EN65e. Midwest Laboratories, Inc, Omaha, NE.

**PRODUCER COLLABORATIONS AT OPEN-SOURCE MONITORING
SITES**

Standard Operating Procedure (SOP) S7

PRODUCER COLLABORATIONS AT OPEN-SOURCE MONITORING SITES

Standard Operating Procedure (SOP) S7

Prepared by:

Richard H. Grant

Reviewed by:

Bill W. Bogan and Albert J. Heber

Effective Date: November 6, 2006

PURDUE APPLIED METEOROLOGY LABORATORY (PAML)

Agronomy, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions	3
4. Health and Safety	3
5. Cautions	4
6. Interferences	4
7. Personnel Qualifications and Training	5
8. Utilities, Equipment and Supplies Provided by Farm Staff	5
9. Procedures	5
10. Data and Records Management	6
11. Quality Control and Quality Assurance	7
12. References	7
Appendix A. Site Information Form for Lagoons and Basins	8
Appendix B. Dairy Corral Site Information Form	10
Appendix C. Producer Event/Change Form	13

1. Scope and Applicability

- 1.1. The collection of high-quality air emissions data from a livestock facility requires communication and collaboration between the field operations staff (FOS) and the livestock producer and his/her staff.
 - 1.1.1. The livestock producer's staff may or may not have a specific interest in the data collected at the open-source site.
- 1.2. This SOP describes the following:
 - 1.2.1. Procedures for collaboration between the FOS and livestock producer and his/her staff responsible for open-source sites (lagoons and corrals)
 - 1.2.2. Information related to the open-source sites that must be collected from livestock producers or his/her staff
 - 1.2.3. Expectations placed upon collaborating livestock producers or his/her staff, and on the FOS
 - 1.2.4. Possible modifications to farm operations that may be needed to accommodate the study.
- 1.3. This SOP applies specifically to the National Air Emissions Monitoring Study.

2. Summary of Method

The research staff must establish and maintain effective communication with any livestock-production that is participating in a research study. The facility design and operational information of the farm must be provided by the producer or his/her staff to the FOS. In addition, the FOS must communicate to the livestock producer and his/her staff those requirements that he/she must meet in order to conduct a successful study. The collaboration and communication will include email messages, phone calls, face-to-face meetings, reports, data forms, presentations, data monitoring, and project documentation such as the Quality Assurance Project Plan (QAPP).

3. Definitions

- | | |
|------------------|---|
| 3.1. Biosecurity | Measures that are taken to prevent spread of disease to or from livestock or poultry. |
| 3.2. DM | Data Manager |
| 3.3. FOS | Field operations staff |
| 3.4. PM/PI | Project Manager or Pincipal Investigator |
| 3.5. PPR | Pull plug with recharge |
| 3.6. QAPP | Quality Assurance Project Plan |
| 3.7. SOP | Standard Operating Procedure |

4. Health and Safety

- 4.1. Beware of, and do not interrupt, worker traffic. FOS may request a slight delay or interruption in normal farm activities during set up and disassembly of instrumentation, but must always defer to the decisions and activities of the producer and his/her staff.

- 4.2. Watch out for vehicular traffic such as litter, feed, animal, milk, and egg trucks, loaders, cars, and other vehicles.
- 4.3. The farm's licensed electrician should conduct or approve all electrical work associated with the study. An example is connecting power to the instrument shelter.
- 4.4. Be careful around animals and birds to prevent personal injury.
- 4.5. Post the phone number for medical emergency personnel and directions (map) to the nearest hospital in the instrument trailer close to the entrance.
- 4.6. Emergency evacuation procedures at each measurement location will be those established by the livestock producer.
- 4.7. Maintain a first aid kit in the instrument trailer.

5. Cautions

- 5.1. Take steps to overcome language barriers (e.g. Spanish vs. English). For example, if any worker has Spanish as his/her primary language, distribute a pamphlet in Spanish indicating the objectives of the study and the importance of taking care of the instrumentation. The pamphlet can be complemented with a video and/or PowerPoint presentation in Spanish that can be distributed to all research sites, and can be included in a periodic (quarterly) refresher session (as rotation of workers in the farms can be high).
- 5.2. Post signs or labels by sensitive test equipment and in measurement paths, indicating that they should not be touched or worked around unless authorized by study personnel. These should also be written in the primary language(s) of the farm workers.
- 5.3. Fence the region around any light emitters that may cause eye damage.
- 5.4. FOS must follow all biosecurity protocols and guidelines requested by individual livestock producer or his/her staff. At minimum, these can be expected to include:
 - 5.4.1. Cleaning research vehicles and equipment entering the farm according to the producer's specific protocol
 - 5.4.2. Accessing only areas that are directly involved in the study
 - 5.4.3. Ensuring that FOS have not exceeded producer-specified limits on foreign travel and/or visits to other poultry or livestock farms.
 - 5.4.4. Notifying the livestock producer prior to visiting the site
- 5.5. The FOS should, to the greatest extent possible, solicit input and suggestions from the livestock producer and his/her staff as to safe locations for equipment, possible hazards that might occur, and appropriate precautions that might best protect equipment and staff.
- 5.6. Remember that the producer's involvement and time commitment to the project is less than that of the FOS. The amount of time required for the livestock producer or his/her staff to execute their commitments to the study is a major factor influencing current and future involvement. Take care not to use more of the producer's and/or staff's time than is absolutely required for the study.

6. Interferences

- 6.1. The participating farms should operate with typical management practices, particularly during active monitoring periods, to minimize the potential for bias in the emission data. If changes to the farm operations occur, they must be documented, and these records retained according to SOP D1.
 - 6.1.1. Livestock producer or his/her staff should notify FOS if research equipment is

- interfering with farm operations and needs to be moved or modified.
- 6.1.2. Livestock producer and his/her staff must agree not to move or modify study equipment themselves.
 - 6.2. FOS must not give tours of the study site without livestock producer's permission. Establish a policy about tours prior to setting up instrumentation on the farm.
 - 6.3. Any add-on research projects at the farm must be approved by the livestock producer.

7. Personnel Qualifications and Training

- 7.1. All livestock producer and research staff involved in the study should read this SOP.
- 7.2. A pre-study training and orientation session for the livestock producer and his/her staff will be conducted by the FOS or Project manager, and will include time for the livestock producer or his/her staff to explain the farm operation. The training session will also be conducted in the primary language of the farm workers, with the help of translated video and/or a PowerPoint presentation.

8. Utilities, Equipment and Supplies Provided by Farm Staff

- 8.1. Personal protection equipment
 - 8.1.1. Levels of protective equipment will likely vary at a given facility, depending upon the season, activity, and other factors.
 - 8.1.2. Protective clothing may be provided by the FOS, or by the livestock producer or his/her staff, depending, for example, on the preferences of the livestock producer. Reach an agreement on this point with each producer at the beginning of the study.
 - 8.1.2.1. For example, if livestock producer wishes to supply their own boots as part of their biosecurity program, FOS will abide by this preference.
- 8.2. A suitable area for placement of the open-source instrument trailer, including all-weather (e.g. gravel) access as necessary
- 8.3. Electric power hookup post (SOPs U4 and U5)
- 8.4. Utility located to ensure the FOS does not accidentally damage underground cables, gas lines, tiles, drains, or other underground hazards while sinking anchors.
- 8.5. Phone line (required) and internet service (preferred) (SOP U4)
- 8.6. A garden hose with a nozzle near the site of the open-source instrument trailer
- 8.7. Restroom and shower facilities, if available

9. Procedures

- 9.1. Livestock producer and his/her staff will allow free access to farm by project staff.
- 9.2. Livestock producer or his/her staff will be apprised of all necessary structural modifications (SOPs U4 and U5).
 - 9.2.1. Provide a project schedule and QAPP to the livestock producer at least 90 days prior to initiation of setup.
 - 9.2.2. Set up a monthly phone call with farm management to solve problems, provide updates and facilitate collaboration.

- 9.2.3. Detailed information about the farm will be collected for each participating site as part of the site-selection process. Site-selection criteria and parameters that should be collected before initiating a study are included as appendices.
 - 9.2.3.1. Lagoon and basin site forms are included in Appendix A.
 - 9.2.3.2. Corral site forms are included in Appendix B.
- 9.2.4. At the end of each measurement period, each collaborating livestock producer at a lagoon or basin site is requested to provide the following information (Appendix A: Table A2) for all barn(s) whose waste goes to the lagoon or basin:
 - 9.2.4.1. Animal inventory, average weight and average age
 - 9.2.4.2. Record of animal movements in and out of the barn
 - 9.2.4.3. Animal diet (feed composition, including N and S levels, and feed consumption rates).
 - 9.2.4.4. Production (quantity and weight of marketed animals, eggs, milk, etc.)
 - 9.2.4.5. Record of sludge removals
 - 9.2.4.6. Record of pump outs (contractor or livestock producer)
 - 9.2.4.7. Record of water consumption
 - 9.2.4.8. Advance notice of any change in production schedules and methods
- 9.2.5. At the end of each measurement period, each collaborating livestock producer at a corral site is requested to provide the following information (Appendix B: Table B2):
 - 9.2.5.1. Animal inventory, average weight and average age
 - 9.2.5.2. Animal diet (feed composition, including N and S levels, and feed consumption rates)
 - 9.2.5.3. Production (quantity and weight of marketed animals, milk, etc)
 - 9.2.5.4. Record of cleaning operations
 - 9.2.5.5. Record of animal movements in and out of the corral
 - 9.2.5.6. Record of water consumption
 - 9.2.5.7. Advance notice of any change in production schedules and methods
- 9.3. Livestock producer and his/her staff should be made aware that placement of equipment or sensors may in some cases require some site modification. FOS and livestock producer staff should work together to ensure that both the equipment and animals are well-protected.
 - 9.3.1. Trailers placed in lots accessible to animals will likely need to be enclosed with protective fencing.
 - 9.3.2. Equipment placed in areas that may occasionally have vehicular traffic may need additional flagging or warning bumpers.
- 9.4. Livestock producer or his/her representative should contact the PM/PI whenever there are any questions or concerns relating to measurement operations, or if there is catastrophic damage to farm operations.

10. Data and Records Management

- 10.1. Site Identification. Each site will be labeled in a standard format to denote site type (in this case, area component), location and number. For example, the finisher swine basin in Iowa is "IA3A". This format is described in more detail in SOP B5. Producers shall not be identified in each site plan.
- 10.2. The FOS will maintain a log of farm visits in the lab notebook.

- 10.3. The livestock producer will maintain a log sheet of non-monitored farm activities that affect emissions (See Appendix C).
- 10.4. Data collected using Producer Event Forms (Appendix C) will be entered into an Excel spreadsheet and included in the site field notes, as described in SOP D1. Management of this data is also described in SOP D1.
 - 10.4.1. Scan all producer event forms, and keep them in electronic form and in paper form at the site. Electronic copies will then be ftp transferred to the PM and DM, as described in SOP D1.
- 10.5. Initial and date any corrections made on hard copies of forms. Use only indelible ink.

11. Quality Control and Quality Assurance

- 11.1. Each FOS member must maintain a log of animal exposures at other farms.
- 11.2. Monthly conference calls ensure communication between FOS and livestock producer.
- 11.3. Livestock producer must check daily to make sure his/her staff are filling out the Producer Event Form, because they do not normally record such data during daily or weekly activities.
- 11.4. Document all decisions and action items on paper in ink during all planning and status meetings so that it can be confirmed that all parties are in agreement.
- 11.5. Be sure livestock producer has a copy of the rationale for the study (found in the QAPP) and a copy of the schedule.
 - 11.5.1. If the livestock producer wishes, provide a copy of any relevant SOPs.
- 11.6. Training/ information session will include a verification of understanding of the basic aspects of the project, importance of the instrumentation and effects of farm operation on the study. Verification of understanding will be through the use of a question and answer session.
- 11.7. Livestock producer should designate a responsible staff member (liaison between the farm and the research team) for collecting and transmitting operational information, and also provide the name of his or her substitute when he or she is not available. This person should attend all informational meetings.

12. References

- 12.1. SOP D1. 2006. Management of Open-source, Weather, and Lagoon-characterization Data. Standard Operating Procedure D1. Purdue Ag Air Quality Lab/ Purdue Applied Meteorology Lab.
- 12.2. SOP U4. 2006. Open-source Instrument Trailer. Standard Operating Procedure U4. Purdue Ag Air Quality Lab/ Purdue Applied Meteorology Lab.
- 12.3. SOP U5. 2006. The Installation of Open-source Measurement Equipment. Standard Operating Procedure U5. Purdue Ag Air Quality Lab/ Purdue Applied Meteorology Lab.

Appendix A Site Information Form for Lagoons and Basins (SOP S7)**Table A1. Lagoon and basin site-selection criteria**

Category	Information
Farm name	
Address, phone	Contact Person:
Livestock Producer	Biosecurity: time since farm visit, days:
	Willing to provide production information?
	Cooperative and enthusiastic participant?
	How long has the farm been in operation under current owner?
	Able to provide feed and water consumption data? Sample analysis records?
	Willing to possibly test abatement technologies after the NAEMS?
	Describe management style (tools used, etc.):
Convenience	Distance from university, miles:
	Phone, internet, and electric power available? 240 VAC?
	Lodging nearby for research team?
Facilities	Generally describe rations:
	Animal breed:
	Describe feed delivery system
	Describe waste collection, handling, treatment and storage systems
	Rate lagoon maintenance from 1 to 5 (1 is cleanest):
	Any emission control measures at facility?
Surroundings	Describe surrounding landscape and topography (hills, flat, etc.)
	Describe other pollution sources within one mile:
	Distance to nearest land application sites
	Distance from public road. Type of road (gravel or paved).

Table A2. Characteristics of test site lagoons or basins.

Descriptive parameters	Unit 1	Unit 2	Unit 3
Livestock type			
Year of facility construction			
Separation distance from barn fans, ft			
Type of storage (basin, lagoon or tank)			
Stage of lagoon (1 st , 2 nd , 3 rd)			
Manure contributors to unit			
Animal 1 type (sows, cows, etc.)			
Animal 1 average weight (lb)			
Animal 1 inventory (# head)			
Animal 2 type (sows, cows, etc.)			
Animal 2 average weight (lb)			
Animal 2 inventory (# head)			
Animal 3 type (sows, cows, etc.)			
Animal 3 average weight (lb)			
Animal 3 inventory (# head)			
Manure collection (flush, scrape, PPR)			
Source flush or recharge water (if any)			
Lagoon loading frequency, hours			
Minimum space surrounding unit, ft.			
Volumetric loading rate, lb/d-ft ³			
Surface loading rate, lb/d-ft ²			
Obstructions within 3X height of unit?			
If yes, what kind? (e.g. trees, barns)			
Height of highest obstruction, ft			
Distance from worst obstruction, ft			
Type of cover (crust, straw, none, etc.)			
Are solids separated from influent?			
Odor control: (digester, additives)			
Sludge removal cycle, years			
Last time sludge removed (e.g., 1999)			
Agitation prior to pumpout?			
Manure removal frequency, days			
Pump out (contractor or producer)			
Type of liner (clay, plastic, etc.)			
Volume, cubic ft			
Surface area, square feet			
Berm slope (e.g. 3:1, 3.5:1, 4 :1, etc.)			
Maximum side length, ft			
Minimum side length, ft.			
Actual freeboard, ft.			
Inner berm height, ft.			
Outer berm height, ft.			
Liquid depth, ft.			
Sludge depth, ft.			
Number of inlets (show on drawings)			
Shape (rectangular, oval, etc.)			

Appendix B Dairy Corral Site Information Form (SOP S7)
(Email to Al Heber, Purdue University: heber@purdue.edu)

Table B1. Dairy Corral Site Selection Criteria, SOP S7.

Category	Information
Farm name	
Address, phone	
Livestock Producer	Provide production and herd management information (DC305)?
	Cooperative and enthusiastic about study?
	Willing to make some nonconsequential changes for the test?
	Willing to train herdspersons about importance of test?
	Able to provide feed and water consumption data per cow group?
	Willing to possibly test abatement technologies after the NAEMS?
	Describe management style (tools used, etc.):
Convenience	Distance to site from university, miles:
	Cell phone signal?
	Phone, internet, electric power available?
	Lodging nearby for research team?
Corrals	Generally describe rations (including forages and purchased feeds)
	Describe feed delivery system.
	Describe waste collection, handling, treatment and storage systems
	Rate corral hygiene from 1 to 5 (1 is cleanest).
Surroundings	Describe surrounding landscape and topography.
	Describe other pollution sources within one mile of site;
Farm	Describe all facilities on the site (# hd, size, age);
	Distance to nearest land application sites.
	On-site heifer growing program?

Table B2. Characteristics of test site: corrals, SOP S7.

Descriptive parameters.	Type 1	Type 2	Type 3
Breed Inventory (cow capacity) Average mass, lb Year(s) of construction Overall site width (E-W dist), ft Overall site length (N-S dist), ft Heifer growing program? Corrals Type (yearlings, lactation, hosp, etc.) Number Corral orientation Corral width, ft Corral length, ft Corral area, ft ² Apron width, ft. Apron length, ft. Apron cleaning (flush, scrape, vac) Maintenance technique (scr, harrow) Maintenance frequency (days) Shade height, ft. Shade length, ft. Shade width, deg. Side slope, % Cross slope, % Cow lane width, ft Cow lane length, ft Windbreak Height, ft Length, ft Lagoon Length, ft Width, ft Depth, ft Berm height, ft Number of stockpiles Weeping pad area, ft ² Number of separators Number of silage bunkers Number of hay barns Parlor Width, ft Length, ft Sidewall height, ft Roof ridge height, ft. Type of ventilation Number of air outlets in ridge Type of sidewall inlets Air circulation fans in ceiling Misting?			

<p>Exit lane drenching? Holding time, min Wash cows? Milking frequency, times daily Parlor occupancy, # cows Holding pen Manure removal (flush, hand wash) Manure removal frequency Width, ft Length, ft Sidewall height, ft Roof ridge height, ft. Type of ventilation (NV, MV) Number of air outlets in ridge Type of sidewall inlets How is feed weighed? How is manure loads weighed? How are animals weighed? Number of pumps to monitor Access around property for sensors?</p>			
--	--	--	--

ON-FARM INSTRUMENT SHELTERS FOR BARN SOURCES

Standard Operating Procedure (SOP) U1

ON-FARM INSTRUMENT SHELTERS FOR BARN SOURCES

Standard Operating Procedure (SOP) U1

Prepared by

Sam M. Hanni and Bill W. Bogan

Reviewed by

Teng-Teoh Lim and Albert J. Heber

Effective Date: November 6, 2007

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability.....	3
2. Summary of Method.....	3
3. Definitions.....	4
4. Health and Safety.....	4
5. Cautions.....	4
6. Interferences.....	5
7. Personnel Qualifications.....	6
8. Equipment and Supplies.....	6
9. Procedures.....	9
10. Data and Records Management.....	11
11. Quality Control and Quality Assurance.....	12
12. References.....	12

1. Scope and Applicability

- 1.1. A trailer is used to house on-site instrumentation (data acquisition hardware, gas analyzers, computer, etc) for collecting barn emission data at a remote farm site.
 - 1.1.1. The On-Farm Instrument Shelter (OFIS) can withstand:
 - 1.1.1.1. Winds of 80 miles per hour
 - 1.1.1.2. Outdoor temperatures from -30 to 110°F
 - 1.1.1.3. Rain falling at rates up to six inches per hour
 - 1.1.1.4. Solar heat gain on the brightest day
 - 1.1.2. The OFIS, structurally, is:
 - 1.1.2.1. 8 ft tall, 7 ft wide and 24 ft long
 - 1.1.2.2. Equipped with windows too small for thieves
 - 1.1.2.3. Locked to prevent unauthorized entry
 - 1.1.2.4. Conducive to conducting tours to visitor and site auditors
 - 1.1.3. Utilities include:
 - 1.1.3.1. High-speed internet
 - 1.1.3.2. Land line phones
 - 1.1.3.3. Electricity to handle up to 100-A load
 - 1.1.4. Barn monitoring can be conducted:
 - 1.1.4.1. With gas sampling locations up to 2,000 ft away
 - 1.1.4.2. With raceway entry to barns up to 150 ft away and 11 ft above the ground
 - 1.1.4.3. From one to five buildings
 - 1.1.4.4. With continuous gas and PM analyzers
 - 1.1.5. The OFIS can facilitate integrated samples of TSP, VOC and odor.
 - 1.1.6. The OFIS provides three lines of lightning protection (Fig. 1) for sensitive instruments.

2. Summary of Method

This SOP outlines the structural specifications for custom-designed mobile trailers for use at barn air emission monitoring sites. The OFIS (Fig. 2) provides protection and proper operating conditions for the data acquisition system and analytical equipment, such as gas analyzers and PM monitors (Fig. 3) and gas sampling systems. It also provides laboratory space for research personnel, space for safe storage and use of compressed gas cylinders for gas analyzer calibrations, and storage for personal protection equipment and clothing used by research personnel. The OFIS is equipped with heating and cooling air conditioning system to provide comfortable working conditions for research personnel and instruments, and sufficient ventilation to remove any noxious gases released from the gas analyzers, as well as filters to capture dust, gases and odors from entering the OFIS. It has facilities for washing hands, and for storing water, food and/or samples. Certain site preparations must be made, as described in this SOP. Once at the site, the OFIS should be supported securely by blocks to avoid putting weight on the tires. Outdoor steps are put in place to gain access to the OFIS. Hurricane straps are used to secure the OFIS to the ground as a protection against storms and high winds.

3. Definitions

3.1. DAC	Data acquisition and control
3.2. DSL	Digital subscriber line
3.3. GFCI	Ground fault circuit interrupter
3.4. HVAC	Heating, ventilation and air conditioning
3.5. O.C.	On-center
3.6. OFIS	On-farm instrument shelter
3.7. PAAQL	Purdue Agricultural Air Quality Laboratory
3.8. QAPP	Quality Assurance Project Plan
3.9. SOP	Standard operating procedure
3.10. TEOM	Tapered element oscillating microbalance
3.11. UPS	Uninterruptible power supply

4. Health and Safety

- 4.1. Exercise proper care when setting up the OFIS, including setting up the blocks supporting the OFIS and make sure that the trailer is properly leveled.
- 4.2. Before pulling the OFIS to or from the research site the trailer's brakes, turn signals and trailer lights should be properly connected to the truck pulling the trailer and checked by the driver to ensure proper function.
- 4.3. Be careful using the outside steps and climbing to the top of the OFIS, particularly in inclement weather. Be mindful of wet or icy steps and ladder.
- 4.4. Ensure that ventilation systems are working properly, taking into account the following points:
 - 4.4.1. Several of the gas analyzers can release noxious fumes. Make sure that these exhausts are properly ventilated outside the OFIS, away from air inlets.
 - 4.4.2. Avoid positioning the OFIS directly in front of the barn exhaust fans, especially large fans.
 - 4.4.3. Clean, maintain, and replace internal air filters frequently to properly mitigate odors and gases. Check the filters at least weekly.
- 4.5. Exercise proper care when wiring the OFIS. Connections must be performed by a certified electrician. Follow all applicable local electrical codes.
- 4.6. During lightning storms, avoid contact with electrical instruments or with the exterior shell of the OFIS.
- 4.7. Be aware of traffic patterns on the farm. Avoid positioning the OFIS in an area that is frequented by trucks or other service vehicles.
- 4.8. Use steps with a landing that is at least 4 ft wide.

5. Cautions

- 5.1. When transporting the trailer to and from the site, make sure that a spare tire for the trailer is available, and that all exterior doors are securely locked. Tow only with vehicles and hitches that are rated for the trailer weight. Follow any applicable state codes, and make sure that the trailer is properly licensed for the state.

- 5.1.1. Do not transport sensitive electronic instruments in the trailer unless they are properly packed and secured.
- 5.1.2. If gas cylinders are to be transported in the trailer, make sure that they are secured as described in SOP G2. Additionally, a strap, fastener, or chain should be used near the bottom of each cylinder to restrain it, and keep it from tipping over. Never transport cylinders with regulators attached (SOP G2).
- 5.2. Flexing of the undercarriage may occur during transportation (specifically, during turns). This may, over time, cause the lug bolts mounting the wheel rims to the hubs to loosen. Check the lug bolts every 100-200 miles during transportation, and tighten as necessary.
- 5.3. Locate the hurricane straps before drilling holes for the heated raceway(s) (SOP U3).
- 5.4. Ensure that the raceway (SOP U3) slopes downward from the OFIS to the barn (3% slope); to keep any raceway moisture out of the OFIS and instruments.
- 5.5. Plug raceway(s) to prevent air flowing through the raceway and into or out of the OFIS.
- 5.6. Make sure all instruments are disconnected before supplying power to the OFIS for the first time. To check that power connections are correct before turning on the power switch use a voltmeter to check that the correct voltage is supplied.
- 5.7. Improper maintenance of the HVAC system can impair performance, leading to uncomfortable conditions.
 - 5.7.1. Check and clean filters weekly and replace filters on HVAC unit when needed.
 - 5.7.2. Check the outdoor coils once a week (at minimum), and clean as necessary.
 - 5.7.3. It is advisable to check the filters and outdoor coils more frequently in summer time, in case the levels of dust and debris are particularly high. If this proves to be true, adjust cleaning schedule as necessary. Clogged air inlets of the HVAC system can impair performance, and may result in damage to the motor, compressor, and/or heating elements.
 - 5.7.4. Do not stack or prop anything against the outdoor condenser unit, or block the outdoor coils in any way.
 - 5.7.5. Do not allow snow to pile up or drift around the outdoor HVAC unit.
 - 5.7.6. Periodically check the permanent drain connection near the inside coil (indoor HVAC unit) to make sure it is unobstructed, and that water is draining properly.

6. Interferences

- 6.1. Heat-generating motors found on the larger pumps required by some of the equipment could place an undue strain on the air conditioning system. Place all pumps in the under-counter storage compartment (Section 8.1.12.1), which is ventilated/cooled by an individual fan.
- 6.2. Extreme weather such as lightning strikes, high winds, and extremely high and low temperatures can affect the OFIS conditions. When these extreme weather situations occur, check the OFIS to make sure it is functioning properly. Set up email alarms for OFIS conditions that are out of limits (SOPs B2); the QAPP will specify to whom such e-mails should be sent.
- 6.3. Flies and dust can enter different equipment and affect the functionality of that equipment. Screen air inlets and exhausts as much as possible.

- 6.4. Rodents can chew through vacuum tubes, insulation and wiring. Use mouse/rat poison, and inspect all tubing frequently for possible damage.
- 6.5. Thieves can be thwarted using secure door and latch locks, and small windows.

7. Personnel Qualifications

- 7.1. Personnel must read and understand this SOP before participating in the setup and/or operation of the OFIS.
- 7.2. Personnel involved in transporting the OFIS trailer must possess the appropriate driver's license(s), and have experience in towing trailers.
- 7.3. Individuals and/or companies involved in the transportations of the trailers should be properly insured.

8. Equipment and Supplies

- 8.1. Trailer: Order or obtain a trailer that meets or exceeds the following minimum specifications, or their equivalents:
 - 8.1.1. Frame & structural support
 - 8.1.1.1. 8" JR I-beam outrigger chassis frame
 - 8.1.1.2. Tandem 6000-lb axles (2 with brakes); springs overslung
 - 8.1.1.3. 10-ply tires
 - 8.1.1.4. Standard towing hitch (welded with 2 5/16" cup and jack)
 - 8.1.2. Floor
 - 8.1.2.1. 2x4 transverse joists (#3 SPF equal or better), 16" on-center spacing
 - 8.1.2.2. 5/8" tongue-and-groove (T&G) plywood deck
 - 8.1.2.3. Standard bottom board (woven plastic insulation support)
 - 8.1.2.4. R-11 faced fiberglass insulation installed tight to warm side of cavity
 - 8.1.2.5. 1/8" vinyl composition tile (Class A/1 F.S.)
 - 8.1.3. Roof
 - 8.1.3.1. Bow trusses at 16" O.C. (30-lb live load), 12" heel x 14" crown 93" long trusses
 - 8.1.3.2. 1/2" gypsum ceiling panel (Class A/1 F.S.)
 - 8.1.3.3. Sheathing: 7/16" OSB 16/0 span index
 - 8.1.3.4. Tie-downs: 2 over the roof hurricane straps
 - 8.1.3.5. Gutter: K-60 J-Rail
 - 8.1.3.6. Roof venting: 1 non-powered screened roof vent
 - 8.1.3.7. R-19 insulation w/vapor barrier
 - 8.1.3.8. 30-gage galvanized steel (Class C/3 F.S.)
 - 8.1.4. Exterior Walls
 - 8.1.4.1. 2x4 studs, 16" o.c., w/top and bottom plates (all 2x4 #3 SPF)
 - 8.1.4.2. Wall height = 8'0"
 - 8.1.4.3. R-11 batt insulation
 - 8.1.4.4. 1/4" vinyl covered paneling (Class A/1 F.S.)
 - 8.1.4.5. 1/8" structural thermoply sheathing
 - 8.1.4.6. 0.019 white aluminum siding and trim
 - 8.1.4.7. 1-1/4" casing (wood grain) base trim

- 8.1.4.8. 1" batten (vinyl) top & corner trim
- 8.1.4.9. 2-1/2" casing (wood grain) door trim
- 8.1.5. Interior Walls
 - 8.1.5.1. 2x4 stud grade SPF, 16" on-center
 - 8.1.5.2. 30" wall height for countertop
 - 8.1.5.3. H.P. laminated counter top
- 8.1.6. Doors
 - 8.1.6.1. Interior
 - 8.1.6.1.1. (1) 24x80 hollow core wood grain pocket door
 - 8.1.6.2. Exterior
 - 8.1.6.2.1. (1) 36"x80" insulated steel with wood jambs and 10x10 safety glazed window, dead bolt standard lock set, and weather stripping threshold
 - 8.1.6.2.2. 25"x25" aluminum open swing compartment doors w/keyed latch
- 8.1.7. Electrical system
 - 8.1.7.1. 100-A load center
 - 8.1.7.1.1. Two single 50-A lines (with grounds) at 120/240 V (single phase)
 - 8.1.7.1.2. External Pullout Switch (with rainproof enclosure)
 - 8.1.7.1.3. Manual Transfer Switch
 - 8.1.7.1.4. 100-A Maximum
 - 8.1.7.1.5. 60 Hz with ground
 - 8.1.7.1.6. Cat # PE 630-V
 - 8.1.7.1.7. 3-wire single phase
 - 8.1.7.1.8. 12 circuit with main 100-A circuit breaker (GETLM812))(+36" AFF)
 - 8.1.7.2. (3) 48" twin-tube fluorescent lights with surface diffusers
 - 8.1.7.3. Incandescent light (1) w/cover in bath
 - 8.1.7.4. Motion-triggered external light (300 W) above the external door
 - 8.1.7.5. (1) 240-V 30-A receptacle dedicated to one circuit breaker
 - 8.1.7.6. (8) 110-V duplex 15-A receptacles
 - 8.1.7.7. (1) 110-V duplex 20-A receptacle dedicated to 1 circuit
 - 8.1.7.8. (2) 110-V 15-A exterior receptacles
 - 8.1.7.9. Wall-mounted power strip, with five pairs of outlets, over the "L" of tables in the clean end of the OFIS, such that this power strip can be routed through the uninterruptible power supply (UPS) (SOP U6)
 - 8.1.7.10. GFCI receptacle (1) by bath
 - 8.1.7.11. Copper Romex 14-gage raceway
 - 8.1.7.12. Manual transfer switch from shore power to generator power
 - 8.1.7.13. Lightning arrestor
 - 8.1.7.13.1. Minimum of 120k Amps/phase capacity
 - 8.1.7.13.2. Titan 120 BP (Part #T120BP120/240Y-R, EFI Electronics, Salt Lake City, UT, or equivalent).
 - 8.1.7.14. Two externally-mounted junction boxes for phone and internet connections
- 8.1.8. Plumbing system
 - 8.1.8.1. (1) 2017 Oval in cabinet lavatory with vanity
 - 8.1.8.2. One 20-gal. tank with inline heater for warming water for washing hands
 - 8.1.8.3. 110-V on-demand water pump for washing hands.

- 8.1.8.4. One 40-gallon holding tank under unit.
- 8.1.9. HVAC system
 - 8.1.9.1. 2.0-ton cooling w/5kW, 240 V electric resistant heat wall hung, return air at unit
 - 8.1.9.2. Blower/filtration system to admit 100 ft³/min of charcoal-filtered and particulate-filtered fresh air. The fresh air exchange rate is 4.5/h.
 - 8.1.9.2.1. This will also create a positive pressure (5-10 Pa) in the shelter with respect to the outside, thus preventing contaminated air from entering the shelter via any cracks which may be present. Pressure differential can be monitored according to SOP A5
 - 8.1.9.3. Exhaust should go out the dirty room to maintain a positive pressure in clean room when pocket door is closed.
- 8.1.10. General exterior
 - 8.1.10.1. Windows: (7) 8" x 14" horizontal slider with aluminum extruded mill frame single obscured glass, screen and white miniblinds
 - 8.1.10.2. Hurricane straps: (2) over roof at front and rear of OFIS
 - 8.1.10.3. Running lights and tail lights
- 8.1.11. Furniture
 - 8.1.11.1. (1) 5' desk w/file cabinet
- 8.1.12. Storage
 - 8.1.12.1.(2) 30" wide x 30" high x 24" deep storage compartment (under the counter) w/exterior access at front of trailer
 - 8.1.12.1.1. Compartment is cooled and ventilated by its own fan to dissipate heat released by motors (Section 9.3.6.1.2) located in the compartment.
 - 8.1.12.1.2. The cooling fans should be equipped with dust filters to avoid PM from the outside being drawn into the compartment along with the cooling air.
 - 8.1.12.1.3. These compartments can, if necessary, be lined with acoustic tiles to minimize noise reaching the interior of the trailer from the motors.
- 8.2. Instrument racks (Fig. 3): These should be constructed of steel, and able to accept sliding trays or rails. Open racks help to keep instrument temperature down and allow air to circulate through easily. All racks should have four wheels, with two of those wheels having locks. There is room for four 88" high instrument racks in the space adjacent to the storage compartment.
- 8.3. UPS (Uninterruptible Power Supply): 120-V In/Out double conversion UPS, with web adapter card and Network Protection Module.
- 8.4. Install two cylinder racks on the wall partition to hold three gas cylinders each. See SOP G2 for the proper means of securing cylinders to the rack(s).
- 8.5. The instrument shelter will have a small refrigerator to store lunches, a microwave for heating refreshments, and a lavatory for washing hands.
- 8.6. Lighting: Light intensity should be 70 foot-candles (750 Lux) in the dirty room and 100 foot-candles (1080 Lux) throughout the clean room.
- 8.7. Water supply of 3 gal/min or higher is needed to clean the HVAC system, with enough pressure and volume to rinse out the coil. This water will be supplied by the farm.
- 8.8. Recreational vehicle ladder mounted at the back of the OFIS, for accessing the roof top to maintain weather-related or PM measurement instruments.
- 8.9. Hitch lock to prevent unauthorized towing.

- 8.10. Spare tires (minimum of one)
- 8.11. Blocks: A sufficient number of concrete blocks to construct six supports, each approximately 16" x 16" and 2 ft high (this height prevents significant weight on the trailer tires). Wood shims should be placed between the trailer and the blocks to help in the leveling of the trailer.
- 8.12. The following general supplies and spare consumables should be kept in the trailer:
 - 8.12.1. Air filters (for A/C inlet, cooling air inlet and trailer circulation air inlet)
 - 8.12.2. Light bulbs and fluorescent tubes
 - 8.12.3. Circuit breakers and/or fuses
 - 8.12.4. Activated charcoal (if charcoal filters are used)
 - 8.12.5. Cleaning supplies: hand soap, paper towels, cloths, bucket, mop, broom, trash bags and trash can
 - 8.12.6. Fly traps or strips, mouse/rat poison
 - 8.12.7. Drinking water and cups
 - 8.12.8. Tools
 - 8.12.8.1. Carpenter's level
 - 8.12.8.2. Hurricane strap anchors

9. Procedures

- 9.1. Transportation
 - 9.1.1. While the trailer is being outfitted (i.e. before it is ready to be towed to the site), keep a hitch lock on the hitch at all times for security purposes.
 - 9.1.2. Before transporting the trailer, verify that all trailer contents have been secured and properly packed.
 - 9.1.3. After attaching the trailer, ensure that the lights and turn signals are properly wired to the towing vehicle, and that they are operational. The verification of the trailer brakes, lights, and turn signals will be conducted by the individual pulling the trailer, and will follow the standard procedures used by the company for which this individual works.
- 9.2. Preliminary site preparation and shelter location
 - 9.2.1. Ensure that there is all-weather access to the OFIS.
 - 9.2.2. Weight the following two factors when determining a location for the OFIS. A compromise between the two may be necessary:
 - 9.2.2.1. The OFIS should be as far away as possible from barn fan exhausts.
 - 9.2.2.2. The OFIS should be as close as possible to the actual sampling points.
 - 9.2.2.3. Line up trailer so OFIS raceway entry is aligned with barn raceway entry.
 - 9.2.3. Orient the OFIS so that the front and back are each within 1" of a line which runs parallel to the barns.
 - 9.2.4. Ensure that the chosen location is accessible to all utilities.
- 9.3. Instrument shelter installation and set-up
 - 9.3.1. After the OFIS is positioned properly, prop the hitch and disconnect it from the towing vehicle.
 - 9.3.2. Level and block up the OFIS.
 - 9.3.2.1. The orientation of blocks should alternate in each course.

- 9.3.2.2. The distance between the three pairs of supports (front, center and back) should be such that the I-beams of the OFIS will fit directly in the middle of the supports. Refer to the blocking diagram provided in Fig. 4.
- 9.3.2.3. Use a carpenter's level and wood shims to level the floor of the trailer.
- 9.3.3. Install electrical services
 - 9.3.3.1. Drive in ground rod next to service entrance panel.
 - 9.3.3.2. Connect the grounding strap to the rod.
 - 9.3.3.3. Connect a protected 100-A switch box to an appropriate circuit breaker or switch box at the farm and to the electrical panel.
 - 9.3.3.4. Ensure that the shelter is properly grounded.
 - 9.3.3.4.1. Test the neutral to earth resistance to determine whether it meets the requirements of the electric code.
 - 9.3.3.4.2. If the value of the neutral to earth resistance exceeds 2 ohms, correct the installation until the resistance is ≤ 2 ohms.
 - 9.3.3.5. Check polarity of all wiring before connecting instruments.
 - 9.3.3.6. Test all lights, air conditioning, heating systems, and fans during the setup phase.
- 9.3.4. Anchor hurricane straps
 - 9.3.4.1. Use both straps at each end of the OFIS
 - 9.3.4.2. Anchors should consist of concrete-filled holes 1-2 ft deep and 6-8 in. wide, into which the straps are set.
- 9.3.5. Drill holes for raceway(s) (SOP U3).
 - 9.3.5.1. If possible (i.e. if elevations of raceways leaving barns permit), the raceways on either side of the OFIS should enter the barn at the same height.
- 9.3.6. Install two telephone
 - 9.3.6.1. The farm or phone company should provide a phone line to the trailer.
 - 9.3.6.2. Run cables into both rooms of the OFIS.
 - 9.3.6.3. Connect and test two phone sets.
- 9.3.7. Install internet connections.
 - 9.3.7.1. DSL cable will be installed and connected by the internet provider.
 - 9.3.7.2. DSL cable is preferred but satellite connection should be used where there is no DSL service.
- 9.3.8. Environmental controls
 - 9.3.8.1. Temperature control
 - 9.3.8.1.1. Maintain shelter temperature within the range of the dew point of the sample air and the highest minimum temperature of the instruments. The appropriate range is 75-90°F
- 9.4. Initiation of operation
 - 9.4.1. Connect and setup the UPS, in accordance with manufacturer's instructions. Verify that the UPS is operating properly. Assure correct polarity of receptacle.
 - 9.4.2. Connect the following instruments to the UPS. Power up each instrument so that all instrumentation in the OFIS is running simultaneously, and verify that this condition can be maintained without tripping circuit breakers.
 - 9.4.2.1. Computer
 - 9.4.2.2. DAC system
 - 9.4.2.3. Gas analyzers

- 9.4.2.4. TEOM controllers
- 9.4.2.5. Do not connect vacuum pumps to the UPS.
- 9.5. Routine operations
 - 9.5.1. Prepare a table of “routine operations”, and post it to the wall of the OFIS. These are duties that must be performed in order to efficiently operate a monitoring site. Each item should have an associated frequency. Included are the following:
 - 9.5.1.1. Check stability of OFIS supports.
 - 9.5.1.2. Check the electrical supply cable and connection box.
 - 9.5.1.3. Check air inlet fan and filter. Verify positive pressure (Step 8.1.9.2.1 & SOP A5).
 - 9.5.1.4. Clean any dust buildup in the pump/motor compartment.
- 9.6. Periodic maintenance
 - 9.6.1. Clean floors as needed.
 - 9.6.2. Clean desks and counters as needed.
 - 9.6.3. Cleaning, recharging, and replacement of the HVAC system should be completed when the system is not properly functioning. Procedures to note when cleaning the HVAC include the following:
 - 9.6.3.1. Use pressurized air or water for cleaning, but do not flatten the fins on the AC. Soaking with warm, soapy water works well.
 - 9.6.3.2. A bathroom brush and shop-vac are suggested cleaning tools.
 - 9.6.3.3. Pull sidegrills off the unit to reach the coils.
 - 9.6.3.4. If light from a flashlight can be seen through all parts of the coils, they are clean.
 - 9.6.4. Replace supply air filter, including charcoal media, as needed.
 - 9.6.5. Inspect the OFIS roof bimonthly. Repair as needed.
 - 9.6.6. Weed abatement (Note: This is expected to be a responsibility of the producer, but may occasionally need to be performed by research personnel).
- 9.7. General cleaning

10. Data and Records Management

- 10.1. Maintain a Site Log with a chronology of events that occur at the monitoring site, and a narrative of problems and their solutions. Items in this log should include the following:
 - 10.1.1. Date, time and initials of person(s) who visit at the site
 - 10.1.2. Brief description of weather
 - 10.1.3. Brief description of any damage to the exterior of the OFIS
 - 10.1.4. Brief description of any changes to the barns and/or production status
 - 10.1.5. Description of work accomplished at the site
 - 10.1.6. Detailed information about the instrument needed for repairs or troubleshooting
- 10.2. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
- 10.3. Manage all data according to SOP B5.
- 10.4. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes.
- 10.5. Initial and date all corrections.

11. Quality Control and Quality Assurance

- 11.1. As described in Section 9.3.3.5, conduct a thorough polarity check on all wiring before connecting any equipment.
- 11.2. An activity sensor will be installed in each OFIS (SOP S3), so that records of site visits by research personnel can be corroborated, and so that unauthorized entry to the OFIS can be documented for future investigation as necessary.
- 11.3. Keep a visitor log book; visitors should sign and date the book.

12. References

- 12.1. SOP B2. 2006. Data Acquisition and Control Software (AirDAC). Standard Operating Procedure B2. Purdue Ag Air Quality Lab.
- 12.2. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 12.3. SOP G2. 2006. Compressed Gas Cylinders. Standard Operating Procedure G2. Purdue Ag Air Quality Lab.
- 12.4. SOP S1. 2006. Producer Collaborations at Barn Monitoring Sites. Standard Operating Procedure S1. Purdue Ag Air Quality Lab.
- 12.5. SOP S3. 2006. Activity Measurements. Standard Operating Procedure S3. Purdue Ag Air Quality Lab.
- 12.6. SOP U3. 2006. Heated Raceway. Standard Operating Procedure U3. Purdue Ag Air Quality Lab.

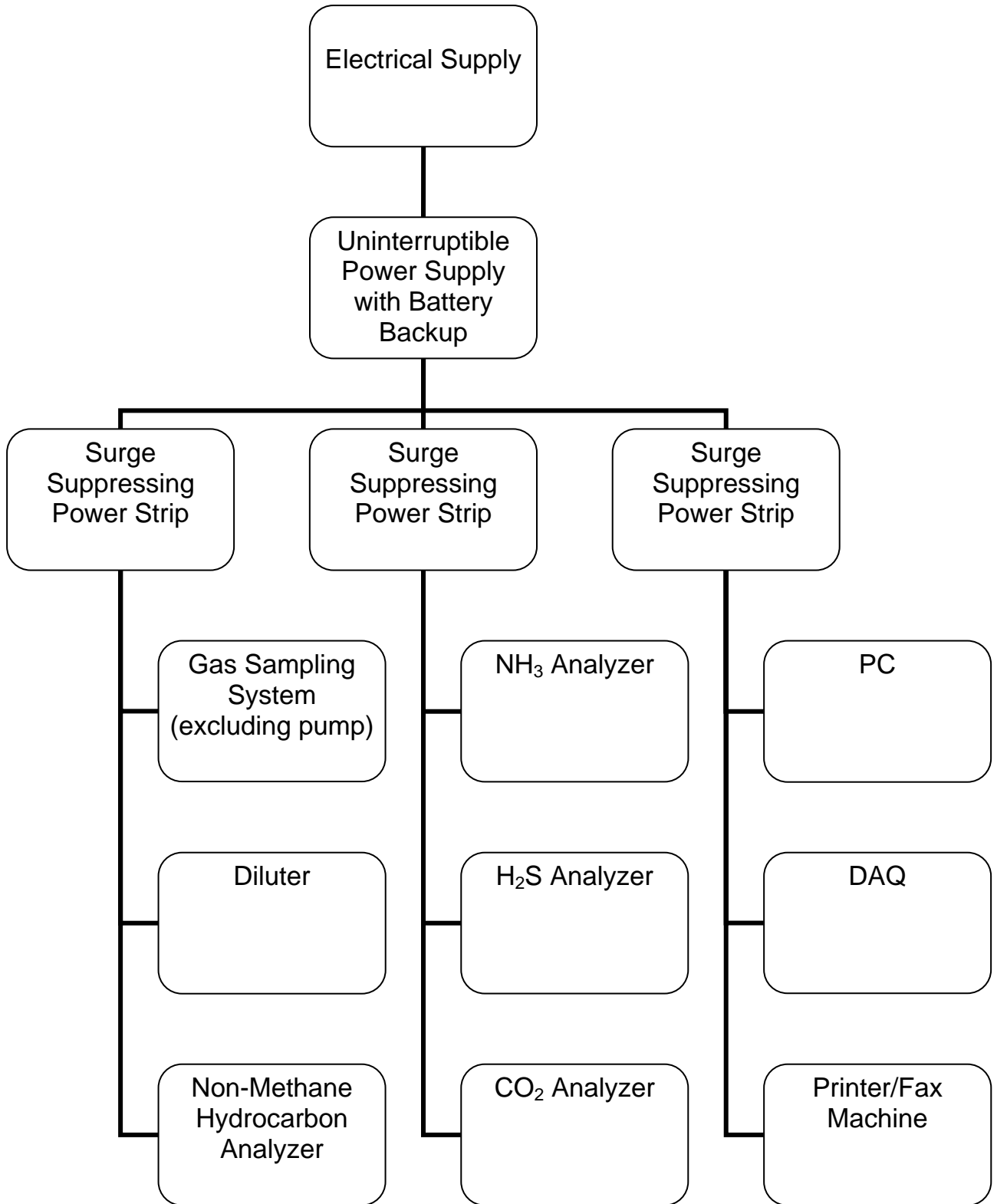


Figure 1. Lightning protection equipment in the OFIS.

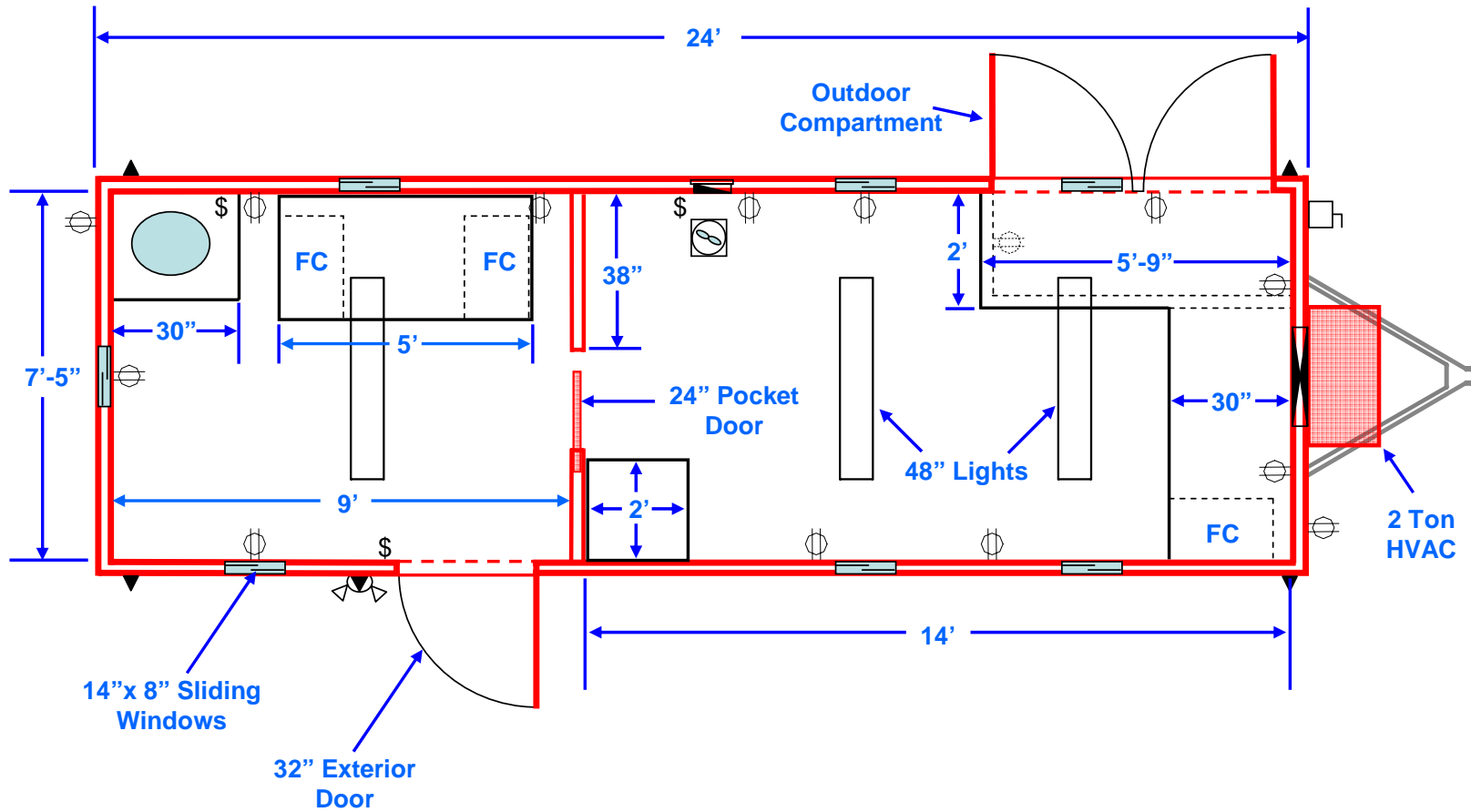


Figure 2. Floor plan of the OFIS. Dirty and clean ends of the lab are left and right of the pocket door.



Figure 3. Instrument racks in the OFIS.

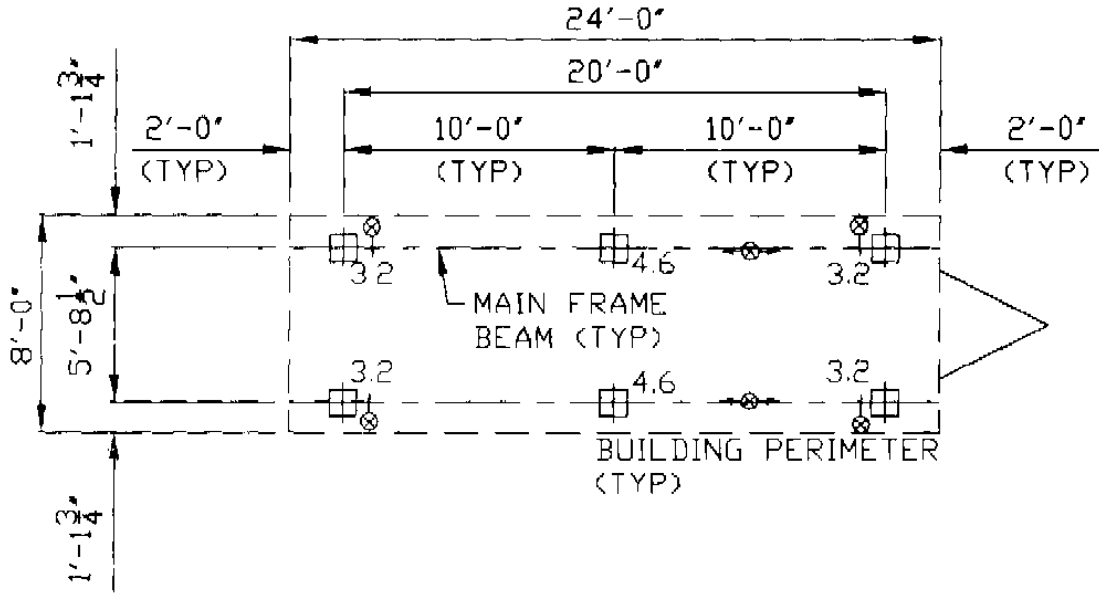


Figure 4. Manufacturer's blocking diagram, showing placement of six supports.

INSTALLATION OF BARN MEASUREMENT TUBING AND CABLES

Standard Operating Procedure (SOP) U2

INSTALLATION OF BARN MEASUREMENT TUBING AND CABLES

Standard Operating Procedure (SOP) U2

Prepared by

Juan Carlos Ramirez, Sam M. Hanni, and Teng Teeh Lim

Reviewed by

Ji-Qin Ni, Bill W. Bogan, and Albert J. Heber

Effective Date: November 6, 2006

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability.....	3
2. Summary of Method.....	3
3. Definitions.....	3
4. Health and Safety.....	3
5. Cautions.....	4
6. Interferences.....	4
7. Personnel Qualifications.....	5
8. Equipment and Supplies.....	5
9. Procedure.....	5
10. Data and Records Management.....	7
11. Quality Control and Quality Assurance.....	8
12. References.....	8
Appendix A. Line Commissioning Form for Installation of Barn Measurements.....	11

1. Scope and Applicability

- 1.1 Monitoring barn emissions requires a large number of sensors, instruments and sampling points. While most of the analytical instruments are located in a centralized on-farm instrument shelter (OFIS), the measurement sensors and air sampling points themselves can be in remote locations of the barns, or inside of the shelter.
- 1.2 The objective of this SOP is to outline the efficient installation of the signal cables, power lines, thermocouple wires, and pressure-sensing and gas-sampling tubing that must be installed between the OFIS and the barns for monitoring gas and PM emissions from Animal Feeding Operations (AFO). This includes the initial preparation of the site layouts which will be used to guide the installation process.
 - 1.2.1. The “power cable” in this SOP refers only to the cable to deliver low-voltage power (maximum 24 VDC) for sensors.
- 1.3 The estimated time to complete the installation ranges from two to six weeks. Progress of the installation should be tracked to ensure that it is proceeding in a timely manner.

2. Summary of Method

This procedure defines the prioritized sequence of activities to efficiently install the instruments, signal and power cables and thermocouple wires, as well as the pressure-, PM- and gas-sampling tubing. The actual sequence will depend on the configuration and limitations of the site. The number of monitored buildings ranges from two to five buildings per site. The procedures outlined in this SOP ensure that the conditions of tubes, signal and power cables meet the requirements stated in the Quality Assurance Project Plan (QAPP) and Site Monitoring Plan(s) (SMP) for the particular project. The method in this SOP provides, through tracking with a Microsoft Project database, appropriate quantitative and qualitative acceptance criteria for determining that the prescribed procedures have been accomplished satisfactorily. The procedures outlined in this SOP also help to verify that the manufacturer-provided specifications for electrical cables and tubing are reviewed for accuracy, noted, and maintained to support the technical requirements of collecting reliable data.

3. Definitions

3.1	GSS	Gas sampling system
3.2	OFIS	On-farm instrument shelter
3.3	PAAQL	Purdue Agricultural Air Quality Laboratory
3.4	PM	Particulate matter
3.5	SOP	Standard operating procedure
3.6	VDC	Volts direct current

4. Health and Safety

- 4.1 Be careful when working with electrical power connections, especially if the barn or pit floor is wet.

- 4.2 Discuss the layout of power cables with the farm's certified electrician for safety and avoid any unnecessary interference.
- 4.3 Wear appropriate personal protective equipment and clothing (SOP S1) while working in the barns.
- 4.4 Be aware of the animals during the installation.
- 4.5 Be careful when working near the ventilation fan(s), as the high-speed fan blades can be a hazard, especially if they are not protected by fan shutters or fan guards.
- 4.6 Be careful when using the outside steps or walkways, and when working on the roof of a barn or OFIS, particularly in inclement weather. Be mindful of wet or icy patches.
- 4.7 During lightning storms, avoid contact with electrical instruments, or with the exterior shell of the barn or OFIS.
- 4.8 Inspect and verify that all moving equipment (e.g. manure scrapers, circulating pumps pits, feeders) in the area where tubing and cables are being installed has been locked out or shut off. Contact the barn manager or electrician to reactivate the equipment after installation.
- 4.9 Use appropriate safety harnesses and/or manlifts when working at dangerous heights such as the open ridge of a naturally ventilated dairy barn.

5. Cautions

- 5.1 Locate the pressure-, PM- and gas-sampling tubing and signal cables in locations that will not affect the daily activities of the farm personnel.
- 5.2 Obtain the farm manager's approval in advance to drill holes on any surface.
- 5.3 Observe the manufacturer's recommended low temperature limits when installing cables. Handling and pulling cables in extreme temperatures can damage the cable sheathing, jacketing, or insulation. To prevent damage of this nature, store the cables in a heated building for at least 24 h prior to installation.
- 5.4 Seal the tubing ends with electrical tape against moisture and contamination (e.g. dust or sand), especially during and after installation in wet locations. Remove the seal only when the tubing is connected to the corresponding instrument.
- 5.5 Remove all abrasive or sharp edges that might damage cables and/or tubing. Examples include sharp edges produced during cutting of pipe sections for raceways (SOP U3), or sharp protrusions on barn surfaces along which cable and tubing are strung.
- 5.6 Minimize the bending of cables and tubing. Follow the manufacturer's recommendations regarding minimum bend radii of cables and tubing.
 - 5.6.1. Install temporary cable supports in manholes, or in places that otherwise will induce a bend radius smaller than the minimum recommended.
- 5.7 Minimize stretching stress of signal cables during and after installation. Do not overtighten cable ties

6. Interferences

- 6.1 One or more barns may be temporarily unavailable for installation because of a scheduled farm activity, such as cleaning or animal removal. Coordinate work with farm personnel for maximum efficiency.

- 6.2 Incorrect labeling of the pressure and air sampling tubes, and signal and power cables can lead to problems. Avoid labels that may fall off, or become illegible. Attach two adhesive labels on each end, at least 1 ft apart. Wrap label onto tubing or cable with multiple (2-4) passes of transparent tape to keep them securely in place.
- 6.3 Minimize exposure of air-sampling tubing to external contamination (water, dust or other material). Seal each end of each tube properly with materials such as electrical tape to avoid exposure during the installation process. Remove the seal only when the tube is ready to be connected to the corresponding filter or gas sampling system (GSS).
- 6.4 Take precautions against farm personnel moving sampling tubing or signal cables, or blocking sensors. Visibly label each sensor's location. Provide contact information for personnel who can answer questions for the farm personnel. Also, inform the farm personnel about the availability of PowerPoint presentation(s) used to educate the producers about the project (SOP S1).

7. Personnel Qualifications

- 7.1 Personnel must read and understand this SOP before participating in the installation.
- 7.2 Personnel should receive training on testing power and signal cables with voltmeters.
- 7.3 Line installers must be able to climb, and maintain their balance while working on ladders or other structures.
- 7.4 Personnel should be able to lift at least 50 lb.

8. Equipment and Supplies

- 8.1 Pressure- and air-sampling tubes (3/8" Teflon tubing) (length to be determined in Section 9.1.3)
- 8.2 Signal cables (varying low-gage multi-conductor cables, shielded if possible) (length to be determined in Section 9.1.3)
- 8.3 Type T thermocouple extension wires (SOP E1) (length to be determined in Section 9.1.3)
- 8.4 Power cables (length to be determined in Section 9.1.3)
- 8.5 Cable ties
- 8.6 Plastic ties
- 8.7 Adhesive paper cable labels and transparent tape
- 8.8 Drill
- 8.9 Electrical tape
- 8.10 Filter holder (Part 6-47-6, Savillex Corporation, Minnetonka, MN) with Teflon membrane filter (47-mm, 1.0- μ m pore size)
- 8.11 Microsoft Project software, and a site-specific schedule file (provided by PAAQL)

9. Procedure

- 9.1 Preparation of drawings and site layouts for each barn (to be conducted by PAAQL personnel)

- 9.1.1. Prepare the monitoring diagram to determine the specific routes for the pressure and air sampling tubing, thermocouple wires, and signal and power cables.
 - 9.1.2. Verify the best layout for all tubing and cables.
 - 9.1.2.1. For air sampling tubing, avoid unnecessary pressure drops and minimize potential interference from farm personnel and machinery.
 - 9.1.2.2. For signal cables and thermocouple wires, avoid placing near power lines.
 - 9.1.2.3. Protect the cable or tubing as much as possible from animals and rodents.
 - 9.1.2.4. Confirm that all of the installed lines and cables will not interfere with the farm's maintenance activities.
 - 9.1.2.4.1. Obtain the farm manager's approval for the layout. If possible, solicit the participation of the operator and/or maintenance (mechanical or electrical) personnel when developing the layout.
 - 9.1.3. Once these layouts have been finalized, use them to determine the total lengths of sampling tubing, thermocouple wires, and signal and power cables that will be required for each site.
 - 9.1.3.1. Estimate the specific lengths for each pressure- or air-sampling tube, and for each cable, from the monitoring diagram or site layout.
 - 9.1.3.2. Sum these lengths to determine the total length required for the site. Obtain enough tubing and cable for the entire layout, including 10% excess to ensure that proper extensions can be made.
 - 9.1.3.3. Include an extra length (about 3 m long) of tubing or cable sampling and sensor ends, to allow any repositioning of the sampling or monitoring location.
 - 9.1.4. Prepare labels for all pressure- and air-sampling tubing and cables. There should be four labels for each, two on each end.
- 9.2 Installation of heated raceway(s)
- 9.2.1. After completing the placement and setup of the OFIS (SOP U1), install the raceway(s) according to SOP U3. Do this before installing tubing and cables, since these will usually need to go through the raceways.
 - 9.2.2. Make sure raceway interiors are clean before installing cables and tubing.
- 9.3 Tubing handling, protection and installation
- 9.3.1. Make sure there is no moisture or dust in the tubing before starting sampling.
 - 9.3.1.1. If contaminated with moisture, flush the interior of the tubing with dry air for a minimum of 5 min, preferably with the tubing extended horizontally.
 - 9.3.1.2. If contaminated with dust, PM or debris, flush with water for 5 min, and then air for 30 min.
 - 9.3.2. Use a suitable feeder device to protect and guide the tubing or cables from the reel into the raceway.
 - 9.3.3. Uncoil the Teflon tubing correctly when there is no feeder device available. Turn the coil while feeding the tubing. Do not pull the tubing from the coil when the coil lays flat on the ground (Figure 1).
 - 9.3.4. Identify every tube or cable with two labels on each end, separated at least 1 ft apart.
 - 9.3.5. Place a filter holder and membrane filter (Section 8.10) at the inlet end of each air-sampling tube, and before the connection to the GSS. This prevents significant PM from entering the air tubing, GSS, and gas analyzers.



Figure 1. The correct (left) and incorrect (right) way to uncoil Teflon tubing when a feeder device is not used.

- 9.4 Installation of thermocouple extension wires, and power and signal cables
 - 9.4.1. Inspect the thermocouple wires and cables for any significant damage.
 - 9.4.2. Install and connect the thermocouple wire and signal cable according to the corresponding SOPs.
 - 9.4.3. Only use cable ties to loosely bundle the cable runs for ease of segregation and identification to various work areas.
- 9.5 Troubleshooting
 - 9.5.1. Air sampling tubing failure
 - 9.5.1.1. While passing air through the tubing with a pump, use rotameters or similar device to measure airflow rate at both ends. If the two flow rates do not agree, inspect the tube for damage or leakage.
 - 9.5.1.2. If tubing was damaged or chewed through by rodents, change out the section and connect using a union. Flush the whole tube with pressurized clean air, and inspect for trapped particles. Measure flow rates again to guard against leakage.
 - 9.5.2. Power or signal cable failure
 - 9.5.2.1. Verify the labels on each end of the cable.
 - 9.5.2.2. Verify that the cable is not broken. Use a multi-meter to test continuity. One easy procedure is to join all the conductors at one end of the cable and test the continuity among the conductors at the other end of the cable. If a discontinuity is found in one or more conductors, follow the cable and check for damage.

10. Data and Records Management

- 10.1 Fill out a commissioning form (Appendix A) for each line installed, stating all the characteristics of the tubing or cable. Store these forms in a three-ring binder, which is kept in the OFIS.
- 10.2 Use a Microsoft Project file to track installation progress. A partial example of this file is shown in Fig. 2.

11. Quality Control and Quality Assurance

- 11.1 Use a single piece of tubing or cable for connection from the sample/measurement location to the OFIS whenever it is possible. Avoid using connector or extensions to the air sampling tubing and cables, to minimize leakage and poor wire connections.
- 11.2 Test and mark the cables for continuity before installation.
- 11.3 Flush all tubing with clean pressurized air to ensure that there are no obstructions, blockages or contaminants inside the lines before putting them into use. Keep one end of the tubing open by temporarily removing the filter while the other end of the tubing is to accept pressurized air so that any particles will be flushed out from the opening end.
- 11.4 Conduct a system configuration check to trace all sampling tubing, and connections to verify the system configuration, and to visually inspect items of equipment to ensure that they are clean, and fit for their purpose prior to undertaking measurement.
- 11.5 Conduct an instrumentation cabling check to ensure that all instrumentation, alarm settings, microprocessor signals and hardwire connections pertaining to the installation are functional. This will also check that signals from the field instrumentation are correctly relayed to the data acquisition and control hardware (SOP B1) and software (SOP B2) and computer displayed in the computer. Record the testing and characteristics of each pressure-, PM- and gas-sampling tube, signal cable and power cable in a Line Commissioning Form, as shown at the end of this SOP.

12. References

- 12.1 SOP B1. 2006. Data Acquisition and Control Hardware. Standard Operating Procedure B1. Purdue Ag Air Quality Lab.
- 12.2 SOP B2. 2006. Data Acquisition and Control Software (AirDAC). Standard Operating Procedure B2. Purdue Ag Air Quality Lab.
- 12.3 SOP E1. 2006. Temperature Measurement Using Thermocouples. Standard Operating Procedure E1. Purdue Ag Air Quality Lab.
- 12.4 SOP S1. 2006. Producer Collaborations at Barn Monitoring Sites. Standard Operating Procedure S1. Purdue Ag Air Quality Lab.
- 12.5 SOP U1. 2006. Barn Instrument Shelter. Standard Operating Procedure U1. Purdue Ag Air Quality Lab.
- 12.6 SOP U3. 2006. Heated Raceway. Standard Operating Procedure U3. Purdue Ag Air Quality Lab.

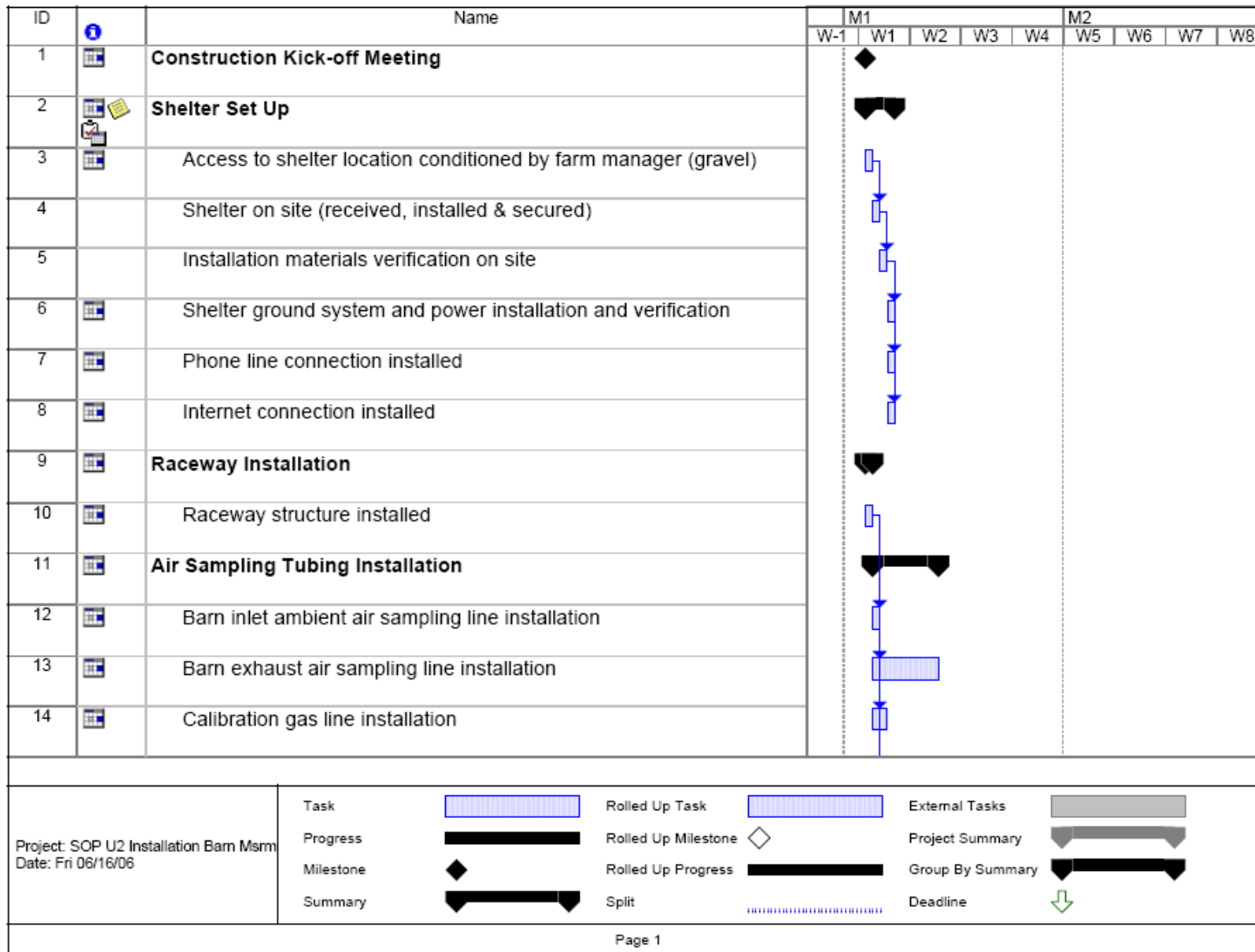


Figure 2. Example of a Microsoft Project schedule for tracking installation of barn measurement lines and cables

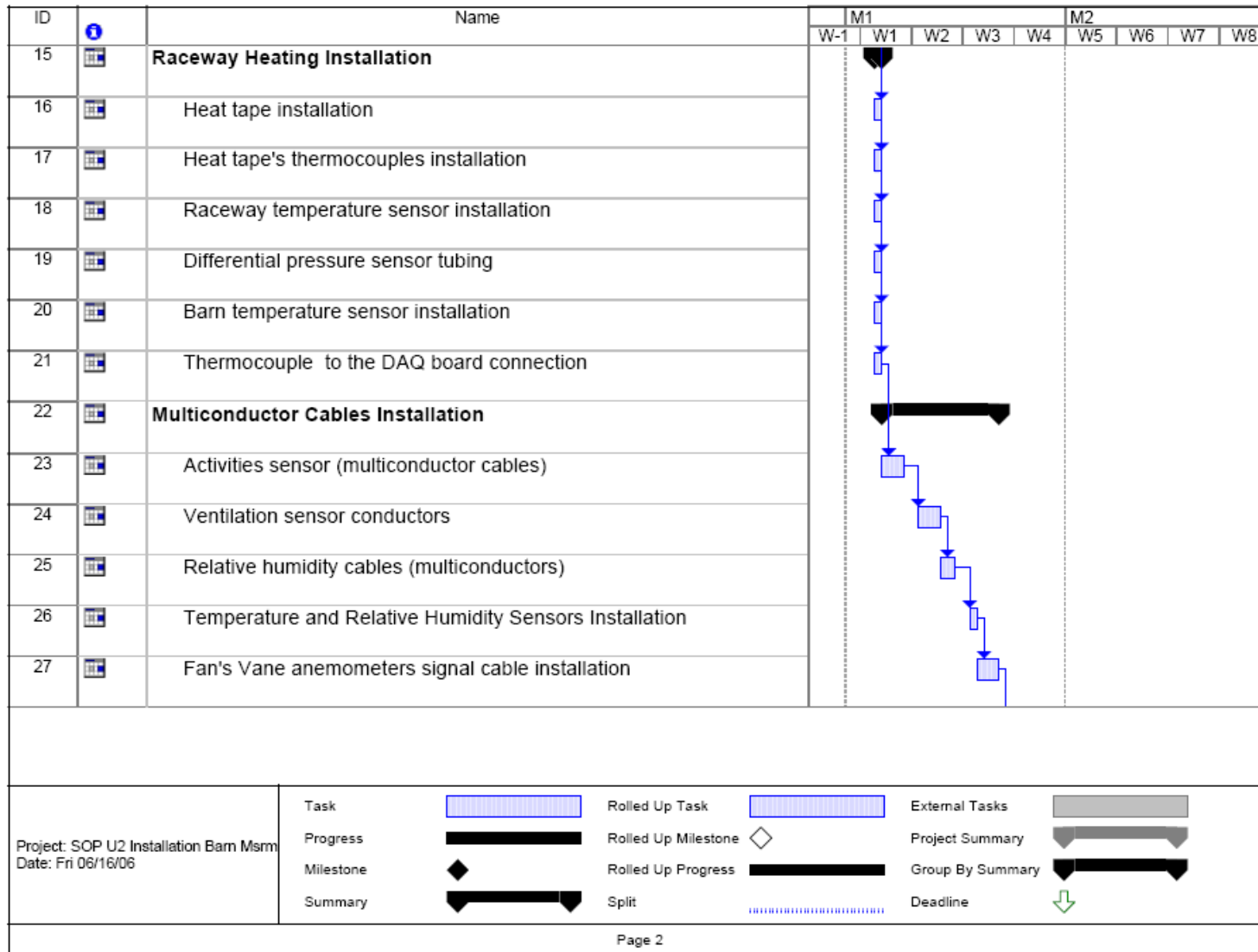


Figure 2 (Continued)

Appendix A. Line Commissioning Form for Installation of Barn Measurements

Line identification description (e.g. farm number/name, barn number, sampling location):

Line Type: Tubing; Signal cable; Thermocouple wire; Power cable
Instrument (if specific to one): _____
Serial Number: _____

Verification of Operation

	Yes	No	Comment
Before installation:			
Installation authorized by farm contact person?			
Farm operator informed of installation?			
Tubing:			
Size and type			
Two labels on each end?			
Tubing cleaned?			
Tubing ends protected from contamination?			
Signal cable:			
Size and type			
Two labels on each end?			
Conductivity of each conductor verified using a multimeter?			
Size and type according to specification?			
Cable ends protected from contamination?			
Power cable:			
Size and type			
Two labels on each end?			
Cable conductivity verified using a multimeter?			
Size and type according to specification?			
Cable ends protected from contamination?			
During Installation:			

Name _____

Date _____

Signature _____

HEATED RACEWAY

Standard Operating Procedure (SOP) U3

HEATED RACEWAY
Standard Operating Procedure (SOP) U3

Prepared by

Teng-Teeh Lim, Erin L. Cortus and Juan Carlos Ramirez

Reviewed by

Albert J. Heber, Bill W. Bogan and Kenneth D. Casey (TAES)

Effective Date: December 28, 2007

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)
Agricultural and Biological Engineering, Purdue University
West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability.....	3
2. Summary of Method.....	3
3. Definitions.....	3
4. Health and Safety.....	4
5. Cautions.....	4
6. Interferences.....	5
7. Personnel Qualifications	6
8. Equipment and Supplies	6
9. Procedure.....	6
10. Data and Records Management	12
11. Quality Control and Quality Assurance.....	12
12. References.....	13
Appendix A: Self-Regulating Heat Tape Connections	14

1. Scope and Applicability

- 1.1. Gas sampling tubes, particulate matter (PM) sampler vacuum hoses, signal wires and power cables running from a barn or other building at an Animal Feeding Operation (AFO) to the on-farm instrument shelter (OFIS) need to be protected from severe weather, workers and vehicles, rodents and other potential hazards.
- 1.2. Condensation inside gas sampling tubes, which could occur if air sampling tubes were exposed to lower temperatures, must be prevented. Condensation control and prevention is critical, because many of the target gases are very soluble in water. Condensation in air sampling tubes can cause significant errors in gas-concentration measurements.
- 1.3. Raceways are heated to prevent condensation in the sample lines.
- 1.4. Raceways connect the OFIS with the building(s) being monitored, and provide the necessary protection for the sample tubes and cables, which are channeled through the raceway into the OFIS.
- 1.5. Raceway lengths range from 1 m (3.3 ft) to 40 m (131 ft); most raceways will be at the short end of this range.

2. Summary of Method

Raceways are most often made from 10 cm dia. (4 in) polyvinylchloride (PVC) pipe, although they can be fabricated from square conduits as well. Removable top covers allow maintenance and addition of new tubing or cables. Drainage holes on the bottom of the raceway prevent any potential condensate buildup or rainwater. The raceways are sloped 2% to 5% downwards toward the barn to prevent water accumulation, and to ensure that any drainage that does occur goes into the barn, rather than into the OFIS. Raceways are usually suspended, but can be buried underground (for example, in areas that are heavily traveled by large vehicles). However, access to the raceway is much more limited if buried. Suspended raceways are generally supported by one or more braces, the number of which depends on the raceway length. Heating tapes, installed along the raceway length, prevent condensation. The self-regulated heat tape is controlled by the data acquisition and control software, AirDAC (SOP B2), with a backup thermostat (normally open contact) to ensure heating in case of PC failure. A capillary tube thermostat (normally closed contact) protects the circuit from overheating. An example of a suspended raceway is shown in Fig. 1.

3. Definitions

- | | | |
|------|--------|---|
| 3.1. | AirDAC | Air quality Data Acquisition and Control software |
| 3.2. | AFO | Animal feeding operation |
| 3.3. | OFIS | On-farm instrument shelter |
| 3.4. | PAAQL | Purdue Agricultural Air Quality Laboratory |
| 3.5. | PM | Particulate matter |
| 3.6. | PVC | Polyvinylchloride |
| 3.7. | QAPP | Quality Assurance Project Plan |
| 3.8. | SMP | Site Monitoring Plan |
| 3.9. | SOP | Standard operating procedure |



Figure 1. Typical configuration of the OFIS and raceways leading into two barns.

4. Health and Safety

- 4.1. If a raceway is high enough that it must be accessed using a stepladder, exercise care when using the ladder. Use ladders only on firm, dry, level ground.
- 4.2. Be careful when working with electrical power connections. Use only non-metallic ladders. The heating tape has power cords carrying 120 V. If possible, disconnect the power to all electric cables in the raceway before working on the interior of the raceway.
- 4.3. Do not work on the raceway if it is raining, especially when there is lightning.
- 4.4. Use gloves when handling cables, to avoid cuts from sharp edges.

5. Cautions

- 5.1. Obtain advance approval to drill holes on any barn surface. The producer should provide the raceway opening (or approve its being made by research personnel) (SOP S1).
- 5.2. Prevent condensation in sampling tubes due to under-heating in cold weather, or when sample tubing is exposed to the cold air inside the air-conditioned OFIS in summer, through the use of heating tapes.
- 5.3. A 30 mA ground-fault circuit interrupter may be required in some installations. Use a heat sink with solid-state relays. Always install a cover over the relay, since the exposed 120 V terminals on top of the units constitute a safety hazard. Consult local fire codes for local requirements.
- 5.4. Provide drainage holes at the bottom of the raceway.
- 5.5. Prevent overheating by using self-regulated heating tape, with a backup thermostat.
- 5.6. Clean the raceway before installing cables and tubing. Debris and sharp objects in raceway could damage the exterior cover of the cables and air tubes.
- 5.7. Remove or cover all abrasions or sharp edges that might damage the tubing or cables.

- 5.8. Seal both ends of the raceway with spray insulation foam or equivalent before starting sampling. Otherwise, the raceway could funnel air between the barn and the OFIS, due to pressure differences between the barn and the OFIS.
- 5.9. Install temporary cable supports at the entrance of the raceway, and in places that otherwise would induce a bend radius smaller than the minimum recommended radius of the tubing.
 - 5.9.1. Recommended minimum bend radius for 1/4" ID (3/8" OD) tubing is 6.5 in.
 - 5.9.2. Recommended minimum bend radius for 1/8" ID (1/4" OD) tubing is 3.5 in.
- 5.10. While feeding tubes through the raceway, seal each end of the tubing to keep contaminants (water, dust or other material) that may be inside the raceway from entering the tubing.
- 5.11. Ensure zero stretching stress of signal cables after installation; make sure there is slack remaining in the cables.
- 5.12. Do not overstress signal cables by over-tightening cable ties.
- 5.13. To avoid the risk of electric shock, insulate connection points on heat tape control relays with heat shrink, electrical tape, or equivalent.
- 5.14. To keep the current draw below the circuit breaker maximum, and to ensure the heat tape provides the rated output, avoid using lengths longer than the recommended maximum length for that cable wattage (Table 1).

Table 1. Maximum heat cable length based on current draw and voltage drop (Nelson Heat Tracing Systems, 2001 and 2004).

W/ft	Maximum Length of Cable (ft) at 50 °F Start-up Temperature (15-A breaker unless noted)
3 (CLT cable)	221
5 (CLT cable)	178
8 (CLT cable)	142
10 (LT cable)	115 (15A breaker), 150 (20A breaker), or 180 (30A breaker)

6. Interferences

- 6.1. Traffic around the OFIS could damage the raceway if the raceway is improperly positioned. Consult with the producer to determine a safe position and height.
- 6.2. Strong winds can affect the stability of a raceway, especially long raceways. Try to avoid positioning long raceways in areas where strong winds will blow perpendicular to the raceway axis. If this cannot be avoided, make sure the raceway is well-supported.
- 6.3. Rodents can enter the raceway and damage cables or tubing. Seal each end of the raceway with spray insulation foam or equivalent after cables are in place to block access, and check the seals periodically for signs of rodent damage. If traces of rodents are seen, add moth balls to the raceway.
- 6.4. The hole where the raceway enters the barn can be a source of cold air that can come in contact with tubing, causing condensation. Seal the junction (Section 9.2.5), and extend the heat tape a minimum of 12 inches into the barn to avoid it.

- 6.5. Cool air entering the trailer (e.g. air conditioner diffusers) can cause condensation if it directly contacts uninsulated, unheated tubes. Tubes inside the trailer can be bundled, insulated and heated to avoid condensation.

7. Personnel Qualifications

- 7.1. Personnel must read this SOP before installing or maintaining raceways.
- 7.2. Personnel should be trained to use the thermostats, interpret the thermocouple output signal, and check for condensation in the air sampling tubes
- 7.3. Personnel must be able to climb and maintain balance while working on ladders, or other structures, and should be comfortable with heights.
- 7.4.

8. Equipment and Supplies

- 8.1. PVC or metallic tubing (PVC is preferred), typically 10 cm (4 in.) diameter
- 8.2. Circular saw
- 8.3. Piano hinges with clasps, pipe clamps, or equivalent hardware to secure raceway cover
- 8.4. Supporting pole(s) and brace(s), if the raceway is longer than 1.5 m (5 ft)
- 8.5. Self-regulating heat cable, W/ft rating at 120 V dependent on site conditions, metallic braid, max. length 50 m (170 ft)
- 8.6. Two thermostats (Sunne 4MY93, or equivalent)
- 8.7. One solid state relay (Watlow SSR 240-25A-DC1, or equivalent) with heat sink and cover
- 8.8. Cable straps and cable ties
- 8.9. Cable and tube supports
- 8.10. Self-sealing pipe insulation, 3/8-in. thickness, diameter depends on the number of air tubes (usually 1 to 1.5 in for about 10 sampling tubes of 3/8" outer diameter)
- 8.11. Cans of spray insulation foam
- 8.12. Moth balls
- 8.13. 30-mA ground fault circuit interrupter (may be required in some installations). The circuit interrupter may be in the form of a circuit breaker or a stand-alone unit installed between the power supply and heat tape power connection.

9. Procedure

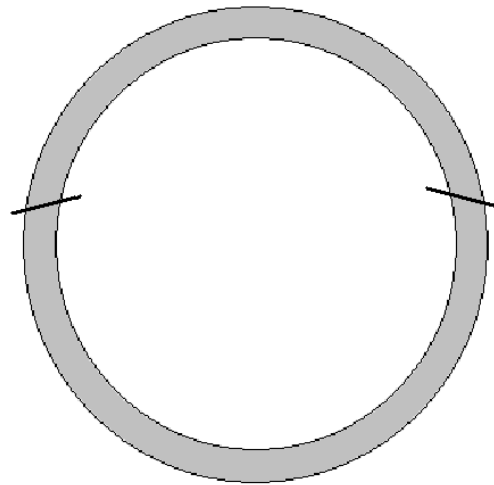
- 9.1. Pre-installation fabrication
 - 9.1.1. Using 10-ft sections of PVC pipe or square conduit, mark a distance of approximately 6 in. away from either end of the pipe.
 - 9.1.2. If using pipe, cut down 1/3 of the diameter of the pipe (Fig. 2A). If using square conduit, cut down approximately 1/3 of the height of the conduit.
 - 9.1.3. On each side of the pipe or conduit, cut out the space between the two original cuts, to form a removable "cover" section. If using circular pipe, make this cut at a slight uphill angle (Fig. 2B), to prevent water from flowing into the joint between the lid and main section. If using square conduit, make the cut flat.

9.1.4. Prepare raceway cover using one of the following methods:

9.1.4.1. Attach piano hinges to the lid and base, approximately every 2-3 ft., to allow opening and closing of the cover.



(A)



(B)

Figure 2. Cutting of circular PVC pipe to fabricate removable raceway covers (A), and the approximate locations and angles for the cuts (B).

9.1.4.2. Cut a length of thin-wall sewer pipe of the same diameter as the raceway, slightly longer than the raceway opening. Remove a section of the sewer pipe (approximately one-third of the pipe circumference) so that the sewer pipe cover can slide over the raceway opening (Fig. 3). Secure cover with pipe clamps.



Figure 3. Using a cut piece of thin-wall pipe as a raceway cover.

9.2. Raceway Installation

- 9.2.1. After the OFIS has been installed and secured (SOP U1) in a location that lines up the OFIS raceway opening(s) with the desired barn raceway opening location(s), measure the distance(s) from the OFIS wall(s) to the wall(s) of the barns, considering that there should be a downward slope (1% to 2%) from the OFIS to the barn (Fig. 1). The barn opening needs to be lower than the OFIS opening.
 - 9.2.2. At the low end(s) of the raceway, drill 2 to 3 small holes (approximately ¼-in. diameter) on the underside of the raceway so that, in the event some water does enter the raceway, it will drain out.
 - 9.2.3. Create the opening for raceway entrance(s) into the barn(s) (SOP S1).
 - 9.2.4. Determine if the raceway requires one or more intermediate supports.
 - 9.2.4.1. If the raceway requires intermediate support, install a support surface at the end of each supporting pole or brace.
 - 9.2.4.1.1. Use semi-cylindrical support surfaces for cylindrical raceways.
 - 9.2.4.1.2. Use flat support surfaces for rectangular raceways.
 - 9.2.4.2. Drive the supporting pole into the ground (at least 2 ft deep), and attach earth-anchored guy wires on both sides, if needed. An alternative method is to install a concrete foundation for the supporting poles.
 - 9.2.5. Thoroughly seal the raceway entrance into the barn (the space between the outer edge of the raceway and the edge of the hole in the wall) with foam insulation.
- ## 9.3. Installing Tubing and Cables
- 9.3.1. Separate air-sampling tubes from power/signal cables and thermocouple wires, as the latter do not require heating. Bundle the air tubes (Fig. 4) with cable ties, with the heating tape at the center for efficient and more uniform heating (Fig. 5).



Figure 4. Bundled and insulated tubing and uninsulated cables and wires entering the raceway from the OFIS (Photo courtesy L Cai, Iowa State University).

9.3.2. Place the thermocouple wire and thermostat capillary sensors that will monitor the temperature of the sampling-tube bundle before installing tube insulation. Install one thermocouple between the exterior of the tubing group and the insulating material, at the coolest location inside the insulation, which is most likely at the furthest distance from the surface of the heat tape). This temperature measurement will be used to control the heat tape. An optional second thermocouple may be added at another location in the bundle as a secondary raceway temperature measurement (Fig. 5).

9.3.3.

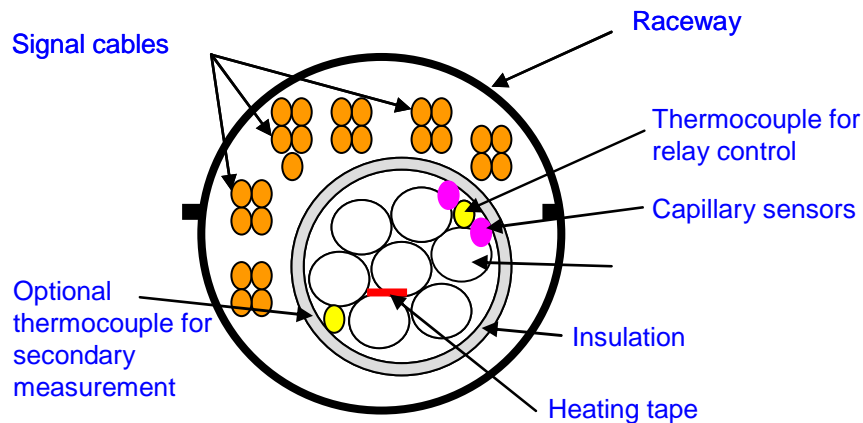


Figure 5. Cross-section of the raceway, showing tubing and signal cables.

9.4. Heating Tape Installation

9.4.1. Install heating tape controls according to Figs. 6 and 7. More information about the heat cable and power connections is provided in Appendix A. The operation of the self-regulating heat tape is activated by the relay contact, which is controlled by the data acquisition and control software, AirDAC (SOP B2). The heat tape operation is also triggered by a backup thermostat (capillary tube) contact (normally open) in the event of data acquisition hardware or software failure (Section 9.3.2). In addition, an overheating protection thermostat (capillary tube) contact (normally closed) is included to prevent any damage to the tubing (Fig. 6). The capillary sensors should be collocated with the thermocouple measurement for relay control.

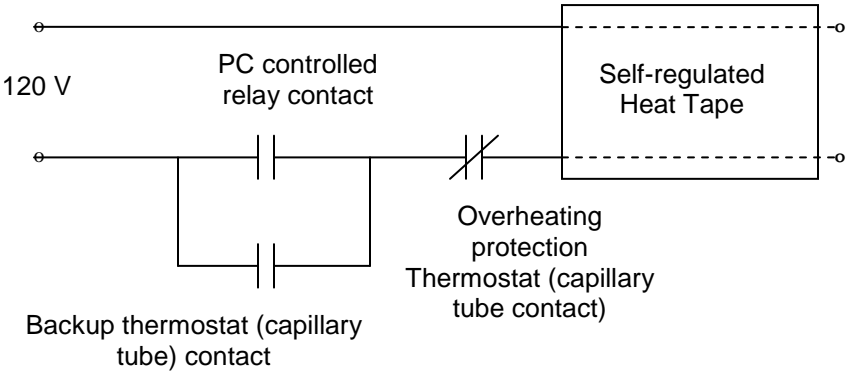
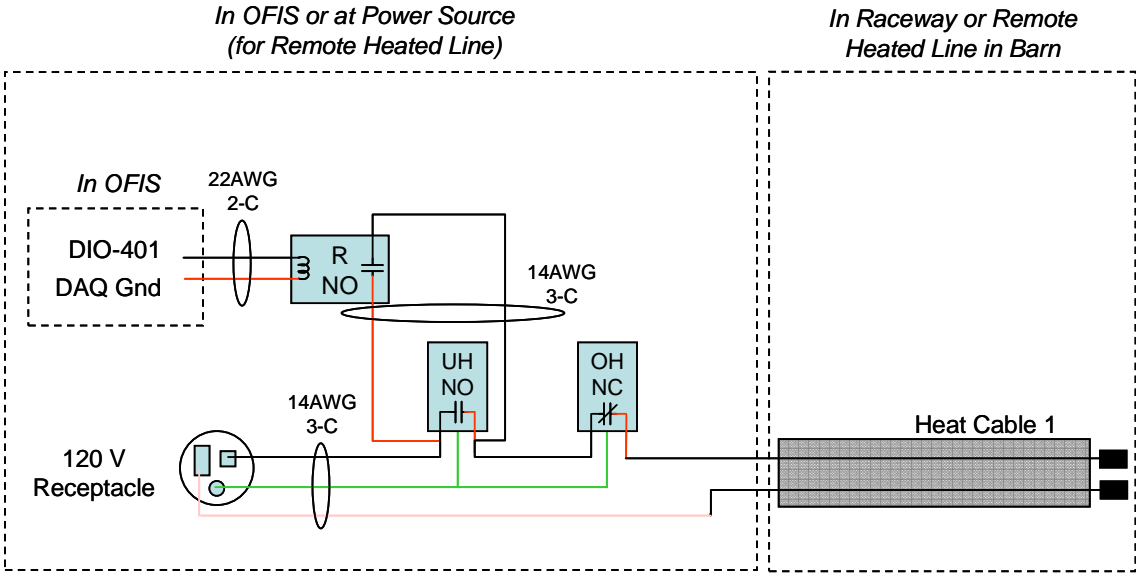


Figure 6. Heating control circuit for maintaining temperatures of air sampling lines above dew point (Heber et al., 2006).



UH: Under-heating thermostat
 OH: Over-heating thermostat
 R: Relay
 NO: Normally open switch
 NC: Normally closed switch
Location in italics

Figure 7. Wiring diagram for heat cable control system.

9.4.2. Heating Tape Operation Check

- 9.4.2.1. Make sure power is supplied to the DAC hardware and heating tape, and that the AirDAC software is operational.

9.4.2.2. The temperature of the air inside each sampling tube needs to be above the dew-point temperature of the sampling point to prevent condensation. To reduce the risk of condensation, a setpoint temperature that is 3 °C above the dry-bulb temperature of the sampling point(s) is recommended (Heber et al., 2006). Actual setpoint temperatures will depend on the sampling point conditions at a site, and are thus site- and season-dependent. Test and review relay and thermostat settings on a seasonal basis. Refer to Table 2 for suggested setpoint temperatures for the relays and thermostats.

Table 2. Heat tape operating setpoints based on sampling point dry-bulb temperature (T_S).

	Setting, °C (°F)	Example Setting, $T_S =$ 30°C
Heat Tape Setpoint (in AIRDAC)	$T_S + 3^\circ\text{C}$ ($T_S + 5^\circ\text{F}$)	33°C
Over-Heating Thermostat Setpoint (Manual Dial Setting)	$T_S + 10^\circ\text{C}$ ($T_S + 20^\circ\text{F}$)	40°C
Under-Heating Thermostat Setpoint (Manual Dial Setting)	T_S (T_S)	30°C

9.4.2.3. Set the temperature setpoint and control deadband in the AirDAC program (under the Data DRC tab) or in the site-specific AirDAC Config spreadsheet for the relay controlling the raceway heat tape. Refer to notes and instructions included in AirDAC for more information on the program's operation and syntax. AirDAC provides an on/off relay control signal based on the lower limit for the raceway temperature at the edge of the tubing bundle, referred to as the setpoint. Below this temperature, the relay will activate (close). As the temperature increases to the setpoint plus the control deadband (sometimes referred to as as "Hysteresis"), the relay will de-activate (open).

9.4.2.3.1. Under the column for the thermocouple controlling the relay operation, set the "Limit: Min" of the measurement equal to the desired setpoint of the raceway. Set the "Hysteresis" or control deadband equal to the desired deadband (suggested value is between 0.5°C and 2°C). Set the "Limit: Max" of the measurement equal to the setpoint plus 5°C. The "Limit: Max" value does not impact the heat tape control, but must be set higher than the "Limit: Min" value to avoid an AirDAC error.

9.4.2.3.2. Under the column for the thermocouple controlling the relay operation, set the value in row 14 within the brackets to {<,DO Control #}, where "<" is syntax that tells AirDAC to use the "Limit: Min" as the controlling setpoint, and DO Control # corresponds to the digital output channel number that controls the relay.

9.4.2.4. To test the heat tape control system, use the suggested setpoint temperatures shown for Test 1 in Table 3 to first test the AirDAC control relay. Observe the heating tape operation and temperature for at least 30–60 min to ensure proper relay operation. If the ambient temperature is similar to the air tube temperature,

increase the relay control set point for faster response. Repeat using the suggested setpoints shown for Test 2 in Table 3 to test the under-heating thermostat. Monitor the raceway temperature to verify that the under-heating thermostat is controlling the heat tape. Repeat using the suggested setpoints shown for Test 3 in Table 3 to test the over-heating thermostat. Again, monitor the raceway temperature to verify that the over-heating thermostat is controlling the heat tape operation. When using a thermostat as the sole control mechanism, a larger temperature variation may be measured because of a larger internal deadband in the thermostat. Reset the backup thermostat and relay setpoints to their normal operating setpoints after completing the tests.

Table 3. Suggested setpoint temperatures for heat tape control system testing.

Test	Suggested Setpoint Temperatures for Tests, °C (°F)		
	Relay	Under-Heating Thermostat	Over-Heating Thermostat
1. for Relay	38 (100)	-18 (0)	49 (120)
2. for Under-Heating Thermostat	0 (32)	38 (100)	49 (120)
3. for Over-Heating Thermostat	0 (32)	-18 (0)	38 (100)

10. Data and Records Management

- 10.1. Record the results of the heating tape operation check in the field notes.
- 10.2. Record any temperature set point changes made to the AirDAC software and thermostat.
- 10.3. Document all data and information on field data sheets, and site logbooks with permanent ink, or in electronic field notes. Initial and date all corrections.

11. Quality Control and Quality Assurance

11.1. Heating System Check

- 11.1.1. The temperatures of the heating tape and air tubes require frequent checking. Personnel maintaining the monitoring site and analyzing data should observe the temperatures to ensure proper operation.
- 11.1.2. If temperatures are not in the correct range, follow the procedures described in Section 9.4.2 to conduct a check of the heating tape and its control system.

11.2. Thermocouple Check

- 11.2.1. Raceway temperature control is used to avoid condensation in gas and PM sampling tubes, and does not require a precision thermocouple calibration. It also does not require that the thermocouples have the same level of accuracy as required in SOP E1. A two-point check (0°C and 30°C) is sufficient (SOP E1). The QAPP will specify how often raceway thermocouples need to be checked/calibrated. If the temperature reading of the raceway thermocouple deviates more than $\pm 3^{\circ}\text{C}$ from that of a NIST-traceable thermometer, replace the thermocouple.

- 11.3. Visually check the air tubes for condensation according to the frequency specified in the QAPP. A weekly inspection is recommended in cold weather.
- 11.4. Perform periodic visual checks of the raceway ends for any traces or rodent damage to the insulation foam. The QAPP for the project will specify the frequency of these checks. Weekly checks are recommended in this SOP.

12. References

- 12.1. SOP B2. 2006. Data Acquisition and Control Software (AirDAC). Standard Operating Procedure B2. Purdue Ag Air Quality Lab.
- 12.2. SOP E1. 2006. Temperature Measurement Using Thermocouples. Standard Operating Procedure E1. Purdue Ag Air Quality Lab.
- 12.3. SOP S1. 2006. Producer Collaborations at Barn Monitoring Sites. Standard Operating Procedure S1. Purdue Ag Air Quality Lab.
- 12.4. SOP U1. 2006. Specification of On-Farm Instrument Shelters. Standard Operating Procedure S1. Purdue Ag Air Quality Lab.
- 12.5. SOP U2. 2006. Installation of Barn Measurements. Standard Operating Procedure U2. Purdue Ag Air Quality Lab.
- 12.6. Heber, A. J., J.-Q. Ni, T.-T. Lim, A.M. Schmidt, J.A. Koziel, P.C. Tao, D.B. Beasley, S.J. Hoff, R.E. Nicolai, L.D. Jacobson, and Y. Zhang. 2006. Quality assured measurements of animal building emissions: Part 1. Gas concentrations. Journal of the Air & Waste Management Association (In Press).
- 12.7. Nelson Heat Tracing Systems. 2004. Nelson Type CLT Self-Regulating Heater Cable, specification/application information. Tulsa, OK.
- 12.8. Nelson Heat Tracing Systems. 2001. Nelson Type LT Self-Regulating Heater Cable, specification/application information. Tulsa, OK.

Appendix A: Self-Regulating Heat Tape Connections

The heat tape consists of the following layers (Fig A1):

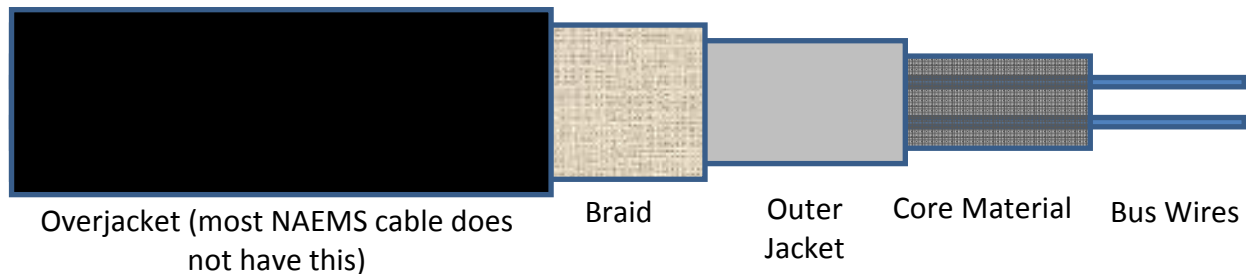


Figure A1. Self-regulating heat tape components.

Important notes when working with self-regulating heat tape:

- The core material acts as a bunch of resistors connecting the two bus wires.
- As current passes through the bus wires, it also passes through the resistors, creating heat.
- The braid is the ground protection.
- There should be no exposure of the core material or bus wires at connection points, as they are both part of the circuit.
- The braid should not contact the core material or bus wires at any connection point or end seal.

Instructions for heat cable connections, and the materials necessary to make one power connection and one end seal are available in the power connection kits. The following information and steps are designed to supplement the power connection kit instructions.

To make a power connection:

1. Remove overjacket. Most NAEMS heat tapes do not have this overjacket.
2. Separate the heat cable from the braid. Create a bulge in braid and use a screwdriver to separate the braid and make a hole to pull the heat cable through (Fig. A2.a).
3. Remove the outer jacket from the heat cable, exposing the black core material (Fig. A2.b)
4. Shave the core material from the sides and remove the core material from the middle (Fig. A2.c).
5. Put heat shrink around the bus wires (Fig. A2.d).
6. Put heat shrink around the exposed core material, keeping the bus wires separated. Pinch the heat shrink closed between the two bus wires (Fig. A2.e).
7. The two parallel conductors of the heat tape can then be connected to the hot wire (black) and neutral wire (white) of the power supply and control system.



(a)



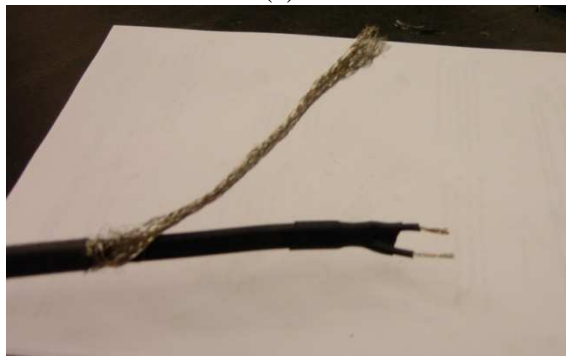
(b)



(c)



(d)



(e)



(f)

Figure A2. Heat tape power connection and end seal procedures. The heat cable shown has a black overjacket (most NAEMS cable will not have an overjacket) and a black outer jacket (most NAEMS cable has a grey outer jacket). The core material is exposed by removing the over jacket and pulling the cable through the braid (a), and removing the outer jacket (b); the bus wires are separated from the core material (c) and covered with heat shrink (d); heat shrink covers the exposed core material (e). When making an end seal, heat shrink covers the exposed core material at the cut end of the cable (f).

To make an end seal:

1. Remove overjacket (if necessary).
2. Push back the braid.
3. Remove a small section of outer jacket.
4. Make a small snip in the core material between the heat cable bus wires.
5. Place heat shrink around the outer jacket and exposed core material, sealing the end (Fig. A2.f).
6. Pull braid over the covered end.
7. Place heat shrink around the end of the cable.

THE POWERWARE 9125 UNINTERRUPTIBLE POWER SUPPLY

Standard Operating Procedure (SOP) U6

THE POWERWARE 9125 UNINTERRUPTIBLE POWER SUPPLY

Standard Operating Procedure (SOP) U6

Prepared by

Bill W. Bogan and Sam M. Hanni

Reviewed by

Albert J. Heber

Effective Date: November 6, 2006

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions	3
4. Health and Safety	3
5. Cautions.....	4
6. Interferences	4
7. Personnel Qualifications	5
8. Equipment and Supplies.....	5
9. Procedures.....	5
10. Data and Records Management.....	7
11. Quality Control and Quality Assurance.....	7
12. References	8
13. Contact Information.....	9

1. Scope and Applicability

- 1.1. A means is needed to prevent power outages or disruptions to the equipment in the on-farm instrument shelter (OFIS) in order to ensure that data collection is not interrupted.
- 1.2. A means is also needed to protect the equipment from possible poor power quality. Power surges or spikes, for example, could be damaging to some of the gas analyzers, or to the DAC system.
- 1.3. This SOP details the use of the Powerware Model 9125 uninterruptible power system (UPS), which provides both of these functions.
- 1.4. SOP U1 provides a list of which equipment should be connected to the UPS. Sampling pumps and converter ovens are not connected to the UPS.

2. Summary of Method

The Powerware 9125 (Powerware Corporation, Espoo, Finland) UPS filters incoming AC power, and provides consistent, spike-free power with a pure sine wave output. The unit's double-conversion online design constantly conditions and controls AC output, assuring zero delay when transferring to backup power in the event of a power failure. The unit incorporates proprietary "Advanced Battery Management" technology, which allows the unit to operate without draining the battery, extends battery life, reduces battery recharge time, and provides a warning when the batteries are nearing the end of their useful life. The Model 9125 can accept up to four Extended Battery Modules (EBMs), further extending the length of time that the UPS can support equipment in the event of a power disruption or outage. Batteries are hot-swappable, so that power to the equipment does not need to be interrupted.

3. Definitions

- | | | |
|------|-------|--|
| 3.1. | EBM | Extended battery module |
| 3.2. | OFIS | On-Farm Instrument Shelter |
| 3.3. | PAAQL | Purdue Agricultural Air Quality Laboratory |
| 3.4. | QAPP | Quality Assurance Project Plan |
| 3.5. | UPS | Uninterruptible power system |

4. Health and Safety


- 4.1. The Powerware 9125 contains potentially lethal voltages. There are no user-serviceable parts in the unit. Do not attempt any service to the UPS other than changing of batteries. Under no circumstances should repairs to the UPS or its batteries be attempted by anyone other than authorized service personnel.
- 4.2. Because the Model 9125 contains batteries as a backup power supply, these voltages may be present even when the unit is not plugged in.
- 4.3. The batteries themselves can cause electrical shocks if they are handled improperly. Do not contact both poles of the battery simultaneously, and do not attempt to service the batteries. Damaged batteries can cause acid burns.
- 4.4. Remove watches, rings, or any other metal objects when handling the batteries.

- 4.5. Use only tools with insulated handles around the batteries.
- 4.6. Do not lay tools or any other metal parts or equipment on top of the batteries, or allow them to contact the battery poles.
- 4.7. When connecting the UPS and EBM, some arcing may occur. This is normal, and will not harm personnel. Minimize arcing by quickly inserting the EBM cable into the UPS battery connector, and establishing a firm connection.
- 4.8. Batteries are lead-acid, and must be properly disposed of or recycled. Do not discard in the trash.
- 4.9. Never unplug the unit while it is turned on, as this will remove the safety ground from the unit, and therefore from all equipment which is connected to the unit.
- 4.10. The UPS and EBM weigh approximately 100 lb combined. Do not attempt to lift the UPS and EBM without assistance.
- 4.11. Operate this UPS only in temperature- and humidity-controlled environments (i.e. inside the OFIS). Do not operate if the temperature exceeds 40°C (104°F), or if the humidity exceeds 95%. Operation under these conditions can present a fire or shock hazard.
- 4.12. Total earth leakage current from all equipment plugged into this UPS must be <1.5 mA.

5. Cautions

- 5.1. If the UPS is positioned vertically, the air vents must be at the top of the unit.
- 5.2. Although EBM batteries will have charged to 80% of their capacity after approximately 2 h in a plugged-in unit, it is recommended that batteries be allowed to charge for 24 h after they are initially installed, or after long-term storage. Batteries may not perform optimally if the power is interrupted to the UPS during this period.
- 5.3. Do not turn the UPS off while it is in Configuration mode (Section 9.2), as this will immediately shut off all power to the equipment as the UPS goes into Standby mode.
- 5.4. Do not plug laser printers into the UPS, as their power requirements are too high.
- 5.5. Make sure the receptacle into which the UPS is connected has the correct polarity. Setup of the OFIS will include a polarity check (with a GFCI receptacle tester) of all outlets.
- 5.6. Disconnect power to the UPS when initially connecting power to the OFIS.

6. Interferences

- 6.1. Batteries in the EBM can wear out. The  indicator and alarm signal that batteries may need to be replaced. If this indicator is lit, press and hold the Test/Alarm Reset button (Section 9.2) for 3 s. If the indicator remains lit, check the battery connections and, if necessary, replace the batteries.
- 6.2. For full battery life, operate the UPS at ambient temperatures as close to 25°C (77°F) as possible. Battery life can be reduced if the unit is operated outside this range.
- 6.3. Dust entering the unit can affect its performance. Keep the surrounding area as dust-free as possible, and vacuum the exterior of the unit periodically if it becomes dusty.

7. Personnel Qualifications

- 7.1. Personnel should be trained in the use of the UPS before installing or operating it.
- 7.2. Each individual must read and understand the Model 9125 manual and this SOP.

8. Equipment and Supplies

- 8.1. Uninterruptible power system (Model, 9125 700-3000 VA, Powerware Corporation, Espoo, Finland)
- 8.2. EBM: (Powerware Corporation, Espoo, Finland)
- 8.3. GFCI receptacle tester (to verify outlet polarity)

9. Procedures

- 9.1. Initial acceptance and installation
 - 9.1.1. Inspect the unit and its box to ensure that no damage has occurred during shipping. Chapter 3 of the UPS Manual describes procedures to file a claim for receipt of a damaged unit.
 - 9.1.2. Check the required battery recharge date on the label of the shipping carton. If the date has passed without the batteries being recharged, do not use the UPS until new batteries are obtained. Contact the distributor (Section 13.2).
 - 9.1.3. Install the Model 9125, and any EBMs, in an instrument rack, following the “Rack-Mount Setup” procedure in the UPS Manual (Pg. 18).
 - 9.1.4. If installing an EBM, plug the EBM cable into the UPS battery connector (Fig. 1). If installing more than one EBM, plug the EBM cable of the second (and subsequent) module into the EBM Battery Connector of the previous module (Fig. 1). See the note in Section 4.1.6 concerning arcing which may occur at this point.

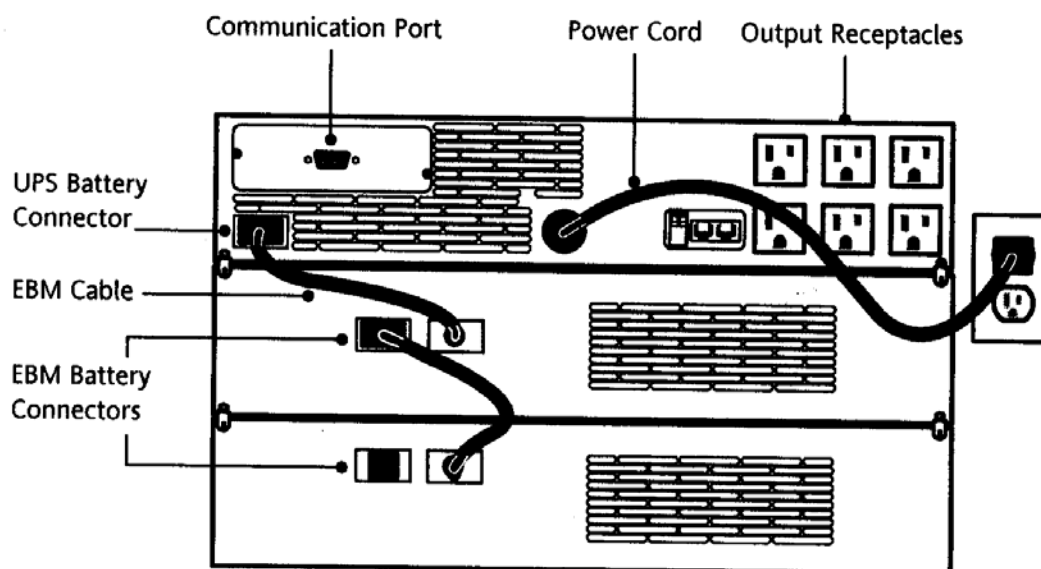


Figure 1. Rear Panel of the Model 9125 and two EBMs, showing connections.

9.2. Operation

- 9.2.1. Connect UPS to power source. It will conduct a 5-s self-test, and enter Standby mode.
- 9.2.2. Turn UPS on. The Normal Mode indicator on the front panel (Fig. 2) will come on, and a bar graph indicator will display the connected load (as percentage of capacity).
- 9.2.3. When the unit is plugged into a working power source (i.e. conditioning power only, not supplying it), it will run in Normal Mode.

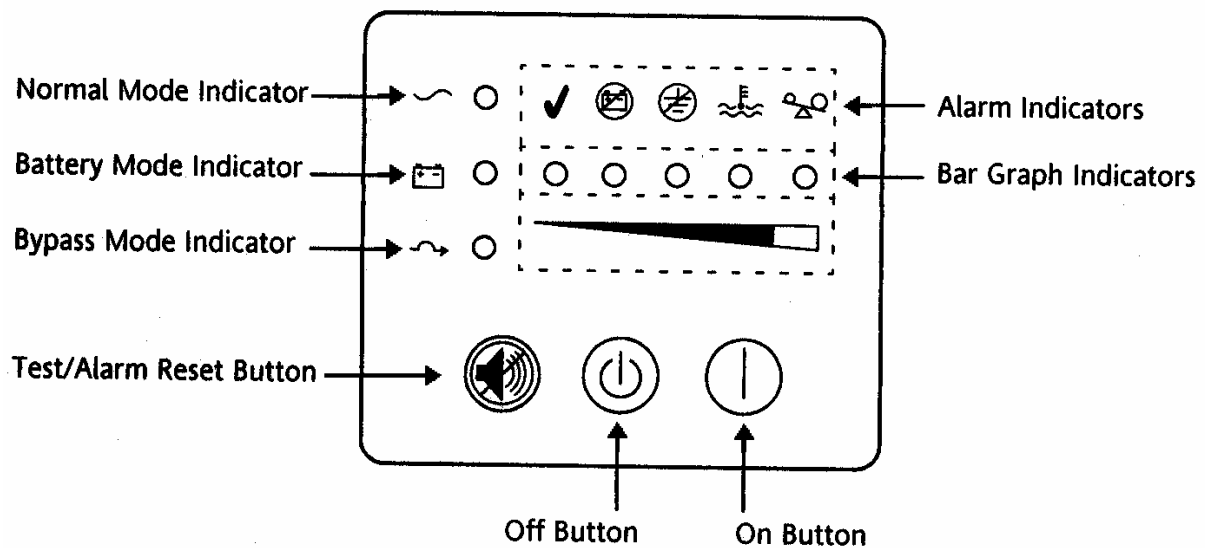



Figure 2. Front panel of the Model 9125, including power buttons and indicator lights.

- 9.2.4. If power to the Model 9125 is disrupted, it will go into Battery Mode. The Battery Mode indicator will be lit, and an alarm will beep.
 - 9.2.4.1. If power is restored before the batteries become drained, the unit will automatically return to Normal Mode, and begin recharging the batteries.
 - 9.2.4.2. If power is not restored quickly enough, the unit will use up its battery power. The  indicator will flash, and a continuous alarm will sound, when there is approximately 3 min of battery power remaining. When the check-mark indicator to the left of the low battery indicator begins to flash, a shutdown is imminent. See Section 11.1.1.
- 9.2.5. The unit will go into Bypass Mode if its temperature is too high, if it detects a fault in its batteries or internal electronics, or if it is overloaded. While in Bypass Mode, the unit continues to condition the utility power source, but the option to switch to Battery Mode in the event of a disruption is not available.

9.3. Configuration

- 9.3.1. 120 VAC input power is the default setting for the low-voltage version of the Model 9125 (the 700-3000 VA), and should not be changed. If it is changed, reconfigure the unit as follows:
 - 9.3.1.1. Press the On and Test/Alarm Reset buttons simultaneously for one beep. The five Bar Graph indicators will either illuminate (option enabled) or not (option disabled). The proper configuration is as follows:
 - 9.3.1.1.1. First indicator (AC Input Failure Alarm): Illuminated/enabled
 - 9.3.1.1.2. Second indicator (Site Wiring Fault Alarm): Illuminated/enabled
 - 9.3.1.1.3. Third indicator (127 VAC Input): Not illuminated/disabled
 - 9.3.1.1.4. Fourth indicator (110 VAC Input): Not illuminated/disabled
 - 9.3.1.1.5. Fifth indicator (120 VAC Input): Illuminated/enabled
 - 9.3.1.2. If any of these are set incorrectly, use the On button to scroll to the correct indicator. The LEDs immediately above the indicators will light individually to denote which indicator is selected. Once an indicator is selected, if that option is disabled, the indicator will switch from not illuminated to flashing.
 - 9.3.1.2.1. Once the desired indicator is selected, change its status by pressing the Test/Alarm Reset button once.
- 9.3.2. The unit will automatically exit Configuration Mode if no changes are made for 2 min.

10. Data and Records Management

- 10.1.1. Record the following in the electronic field notes:
- 10.1.2. Data pertaining to initial setup (receptacle polarity check, earth leakage current)
- 10.1.3. Dates and results of all UPS self-tests
- 10.1.4. Dates and specifics of any changes in configuration
- 10.1.5. Manage the field notes according to SOP B5.

11. Quality Control and Quality Assurance

- 11.1. The Model 9125 provides audible alarms for a number of conditions that might affect the unit's performance. A full list of these alarms is provided in Table 10 (Pgs. 56-57) of the UPS Manual. Included among these are the following:
 - 11.1.1. Warnings indicate when the unit switches to battery power, when the unit has only 3 min of battery power remaining, or when a shutdown is imminent due to low battery power. Under all of these circumstances, switch off all the equipment using the power supplied by UPS. Write down the time and cause of this action in the field notebook, and transfer to the electronic fieldnotes when power is restored.
 - 11.1.1.1. If the total load of all equipment is very close to the maximum of the power circuit, turn off power to most of the equipment, so that there will not be a sudden significant draw when power is restored.
 - 11.1.2. Warnings indicate when the batteries in a plugged-in unit are not charging properly. The last of these can mean that the batteries are not connected properly, or that they need to be replaced. Follow Section 6.1 to run a self-test and address this warning.

- 11.1.3. Warnings indicate when the unit is overtemperature. Turn off and unplug the unit for at least 5 min to cool, check the airflow around the unit, and verify that there is no dust buildup on the vents. If the alarm persists when the unit is turned back on, contact the distributor.
- 11.1.4. Warnings indicate when the unit is overloaded. Turn off and unplug the unit for at least 5 min, unplug some of the equipment from the UPS and restart. Determine if a higher-capacity UPS is required. Fig. 3 shows the pattern in which indicator lights will be lit to display the load level of the UPS.
- 11.2. Even in the absence of alarm messages, perform a self-test (Section 6.1) of the unit on a weekly basis. Record self-tests in the electronic field notes.
- 11.3. The hot-swapping feature of the batteries allows battery replacement without interrupting power to the equipment that is connected to the UPS, thus preserving the continuity of data.

12. References

- 12.1. UPS Manual. 2003. Powerware 9125 Two-in-One User's Guide. Powerware Corporation, Espoo, Finland.
- 12.2. SOP U1. 2006. On-farm Instrument Shelters for Barn Sources. Standard Operating Procedure U1. Purdue Ag Air Quality Lab.

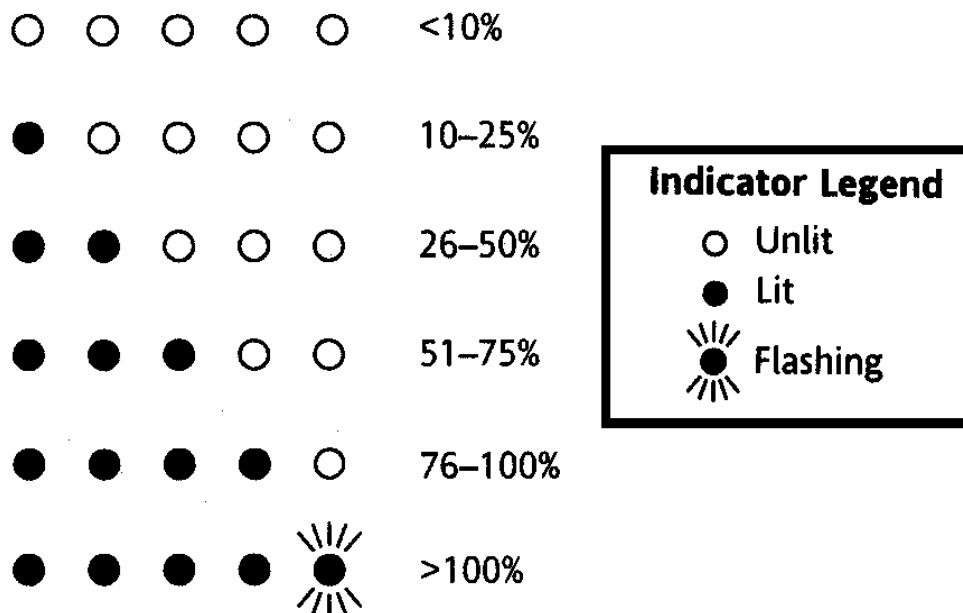


Figure 3. Status of Model 9125 indicator lights in Normal Mode at various loads (% capacity).

13. Contact Information

13.1. Manufacturer:

Powerware Corporation
Koskelontie 13, FIN-02920 Espoo, FINLAND
Phone: +358-9-452 661
Fax: +358-9-452 665 68

13.2. Distributor:

Perfect Power
3405 W. 96th St., Indianapolis, IN 46268
Phone: 317-874-4260
Fax: 219-462-2227

**SAMPLING OF VOLATILE ORGANIC COMPOUNDS (VOCs) IN AIR
SAMPLES THROUGH USE OF SORBENT TUBES
Standard Operating Procedure (SOP) V1**

**SAMPLING OF VOLATILE ORGANIC COMPOUNDS (VOCs) IN AIR
SAMPLES THROUGH USE OF SORBENT TUBES
Standard Operating Procedure (SOP) V1**

Prepared by

Hua Xu and Bill W. Bogan

Reviewed by

Albert J. Heber

Effective Date: November 13, 2007

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	4
3. Definitions	4
4. Health and Safety	5
5. Interferences	5
6. Personnel Qualifications.....	6
7. Equipment and Supplies	7
8. Procedures	7
9. Data and Records Management	9
10. Quality Control and Quality Assurance	10
11. References.....	13

1. Scope and Applicability

- 1.1. This method samples volatile organic compounds (VOCs) at livestock barns onto sorbent tubes.
- 1.1.1. Specifically, this method will use sorbent tubes packed with carbopack C/X to sample VOCs. Compounds which are amenable to this method include phenols, volatile fatty acids (VFAs), alcohols, aldehydes, alkanes, aromatics, amines/amides, ketones, esters, and sulfur-containing compounds, such as those listed in Table 1.

Table 1. Compounds suitable for sampling with carbopack C/X sorbent tubes.

ACIDS	ALCOHOLS	AMINES/AMIDES
Formic acid	Ethanol	Trimethylamine
Acetic acid	Isopropanol	Triethylamine
Propanoic acid	1-Propanol	Benzenamine, N-phenyl-
Butanoic acid	1-Butanol	3-methyl Indole
Propanoic acid, 2-methyl -	2-Butanol	Formamide
Pentanoic acid	Phenol	Acetamide
Butanoic acid, 2-methyl-	Phenol, 4-methyl-	Acetamide, N,N-dimethyl-
Butanoic acid, 3-methyl-	Benzyl alcohol	Caprolactam
Hexanoic acid	Phenol, 4-ethyl	Propanamide, N,N-dimethyl-
Hexanoic acid, 2-methyl-	Phenol, 2,4-bis(1,1-dimethylethyl)-	
Benzoic acid	Butylated hydroxytoluene	Others
1,4-benzenedicarboxylic acid	,5-di-tert-Butyl-4-hydroxybenzyl alcoh	D-Limonene
ALDEHYDE	ALKANES	AROMATICS
Acetaldehyde	Hexane	Benzene
Pentanal	Undecane	Toluene
Hexanal	Dodecane	1H-Inden-1-one, 2,3-dihydro-
Heptanal	Tridecane	
Octanal	Tetradecane	
Nonanal	Pentadecane	
Benzaldehyde	Hexadecane	
ESTERS	S-contain Compounds	KETONES
n-Propyl acetate	Dimethyl disulfide	Acetone
Ethyl citrate	Dimethyl sulfone	Acetophenone
Ethyl Acetate	Thiophene, tetrahydro-, 1,1-dioxide	2-Butanone, 3-hydroxy-

- 1.2. The stated detection limit for target analytes in the sampled air is 0.5 to 25 parts per billion (ppbv) concentration.

- 1.3. This method is generally effective for temperatures between 0°C and 40°C. In general, an increase in temperature of 10 °C will reduce the breakthrough volume (Section 3.1) for sorbent tubes by a factor of 2.
- 1.4. The methodology described in this SOP is derived from USEPA Method TO-17, “Determination of Volatile Organic Compounds in Ambient Air Using Active Sampling onto Sorbent Tubes” and ASTM D6196-03 “Standard Practice for Selection of Sorbents, Sampling, and Thermodesorption Analysis Procedures for Volatile Organic Compounds in Air.”

2. Summary of Method

Prior to use, tubes are conditioned by heating to 350°C for at least 150 min, during which time at least 100 mL/min of pure helium carrier gas is passed through them. The monitoring procedure then involves pulling a known volume of air through specially fabricated glass tubes packed with a chemisorbent matrix. Depending on the specific chemical nature and selectivity of the sorbent matrix, this results in collection of one or more particular classes of VOCs. The collected sample is then transported back to the laboratory, where it is analyzed by gas chromatography/mass spectrometry (GC/MS). The analysis of the sorbed VOCs is facilitated by thermally desorbing them from the adsorption tube onto the focusing trap of the GC/MS system. The analytical component of this overall methodology is covered in SOP V4. This SOP includes guidance on selection of packing material for sorbent tubes, as well as procedures for screening the sampling location by taking single-tube samples to allow estimates of the nature and amount of VOCs in the sample. In this process, two tubes are used to collect, nominally, 3 to 6 L (50 to 100 mL/min for 1 h). The results from these are compared to the method’s performance criteria to determine acceptability of the data. This SOP also describes procedures and requirements for suitable laboratory and field blanks.

3. Definitions

- | | |
|---------------------------------|---|
| 3.1. Breakthrough Volume (BV) | The volume of air (assuming a constant analyte concentration) that may flow through a sorbent tube before a non-negligible percentage (typically 5%) of the analyte passes through the tube without sorbing. |
| 3.2. GC/MS | Gas chromatography/mass spectrometry |
| 3.3. PTFE | Polytetrafluoroethylene |
| 3.4. ppb | Parts per billion |
| 3.5. QAPP | Quality Assurance Project Plan |
| 3.6. RH | Relative humidity |
| 3.7. Safe Sampling Volume (SSV) | A volume of air equal to 2/3 of the breakthrough volume |
| 3.8. SOP | Standard operating procedure |
| 3.9. Sorbent Strength | Describes the affinity of sorbents for VOC analytes. Sorbent strength is generally a function of surface area (although matrix hydrophobicity does play a small role). Sorbents are classified according to the |

specific surface area of the matrix. “Weak” sorbents have a matrix surface area $< 50 \text{ m}^2/\text{g}$. “Medium” sorbents’ matrix surface area is $\approx 100\text{-}500 \text{ m}^2/\text{g}$, and that of “Strong” sorbents is $\approx 1000 \text{ m}^2/\text{g}$.

3.10. VFA

Volatile fatty acid

3.11. VOC

Volatile organic compound(s)

3.12. NIST

National Institute of Standards and Technology

4. Health and Safety

- 4.1. When sampling in areas with known or suspected high levels of VOCs, use personal protective clothing.
- 4.2. Effluent gases from tubes that are undergoing conditioning should be passed through a charcoal filter to prevent desorbed VOCs from polluting the lab atmosphere.

5. Interferences

- 5.1. Sorbent artifacts
 - 5.1.1. Artifacts are typically less than 1 ng/tube for sorbent tubes with well-conditioned graphitized carbon blank sorbents (e.g. carbopack C and carbopack X).
 - 5.1.2. Stringent tube conditioning (Section 8.1) and careful tube handling procedures after conditioning are essential for minimizing these artifacts.
 - 5.1.3. Some charcoal types contain metals that will catalyze degradation of sorbed VOCs during the thermal desorption step. This phenomenon produces artifacts, and results in low analyte recovery, because the analyte is destroyed and converted to one or more breakdown products. Charcoal traps should not be used with this method unless lack of this interference possibility can be assured.
- 5.2. Artifacts from long-term storage of tubes
 - 5.2.1. Literature cited in USEPA Method TO-17 states that the levels of artifacts encountered on tubes (CarbotrapTM, CarbosieveTM, Tenax®) which have been conditioned and stored according to the methods described in this SOP are on the order of 0.01 ng after 1-2 mos and 0.1 ng after 6 mos.
 - 5.2.2. Tubes that have been stored for 2 to 6 mos must be re-conditioned for at least 15 min before use.
 - 5.2.3. Tubes stored for more than 6 mos since conditioning must be fully re-conditioned before they are used.
 - 5.2.4. Because contamination of this sort will also occur as tubes containing sample are stored, all tubes must be analyzed within one month of sample collection.
 - 5.2.5. Contamination of certain other sorbent types – including porous polymers such as Chromosorb 106 can be orders of magnitude higher, even after just after 1 week in storage. Do not use these sorbents under this SOP unless it is feasible to condition them immediately prior to use.

- 5.3. Additional artifacts generated during sampling and sample storage.
 - 5.3.1. Literature cited in Method TO-17 states that artifacts such as benzaldehyde, phenol and acetophenone can be formed via oxidative breakdown of Tenax® when sampling air streams with high concentrations (100-500 ppb) of ozone.
 - 5.3.2. When sampling low levels (<10 ppb) of these analytes with Tenax®-based tubes in any area where appreciable ozone concentrations are either known or suspected, use an ozone scrubber.
- 5.4. Water interferes with sample collection by condensing on the sorbent matrix and blocking the access of the molecules that are intended to be sorbed/trapped.
 - 5.4.1. Never use sorbent tubes that are at a lower temperature than the atmosphere being sampled, as this will promote condensation. Tubes must be at least the same temperature as the airstream being sampled.
 - 5.4.2. Water collection and entrainment within the matrix can be minimized by choosing the most hydrophobic matrix that is appropriate for a particular group of analytes.
 - 5.4.2.1. SSVs of VOCs on hydrophobic matrices (e.g. Tenax® and other porous polymers, Carbotrap™ and Carbopack™) are relatively unaffected by atmospheric humidity. Carbonized molecular sieve-based sorbents (e.g. Carbosieve™ SIII, Carboxen®) are affected by high humidity, and their SSVs should typically be reduced by a factor of 10 at high humidity (90-95% RH). Hydrophilic zeolite molecular sieves cannot be used at all at high humidity.
 - 5.4.3. Water can also interfere with the subsequent analytical steps. Approaches for minimizing this interference are discussed in SOP V4.
- 5.5. Air movement, either outdoors in the form of wind, or indoors as the result of a fan or other ventilation, is not a problem at air speeds less than 10 mph. Above this speed, orient tubes perpendicular to the prevailing wind direction during sampling. Physically shelter tubes from the direct draft if wind speeds exceed 20 mph.
- 5.6. In areas of extremely high particulate concentrations, it may be necessary to connect a particulate “pre-filter” (e.g., a 2-micron Teflon® filter or short clean tube containing a loose plug of clean glass wool) to the inlet end of the tube.
 - 5.6.1. Some analytes, however, may be trapped by Teflon® or glass wool.
 - 5.6.2. Particulates, trapped either on the prefilter or in the sorbent tube, may act as a source or sink for VOCs; if trapped in the tube itself, they may remain there through several cycles of sampling and desorption.
 - 5.6.3. Replace the particulate filter frequently.
- 5.7. Compounds that are too labile or reactive for conventional GC analysis are not suitable for this method.
- 5.8. Inorganic gases such as oxides of C, S, and N (with the exception of CS₂ and N₂O) cannot be sampled by this method.

6. Personnel Qualifications

- 6.1. Personnel should be trained in the use of all applicable instrumentation, and in appropriate sample-handling procedures, before performing the activities in this SOP.
- 6.2. Each analyst must read applicable equipment manuals and this SOP before performing these activities.

7. Equipment and Supplies

7.1. Sampling tubes

- 7.1.1. Some information on the relative strengths and suitable analytes for a range of common, commercially-available sorbent matrices is given in Table 2.
- 7.1.2. Multibed glass tubes packed with 18 mm of Carbopack C 40/60 mesh and 38 mm of Carbopack X 40/60 mesh are appropriate for VOC sampling under this SOP.
- 7.1.3. Tubes must be compatible with the thermal desorption unit (GERSTEL TDS-G or equivalent).
 - 7.1.3.1. Use GERSTEL tubes with an outside diameter of 0.6 ± 0.1 mm, and a length from 117.5 ± 177.8 mm.
 - 7.1.3.2. The position and length of the sorbent beds must be such that no part of the bed extends outside that tube portion directly heated by the thermal desorption oven.

7.2. Sampling pump

- 7.2.1. Must be capable of operating from 10 to 200 mL/min. Should be able to deliver a constant flow rate for a wide range of tube impedances. This allows the pump to compensate for moderate impedance variations between different sorbent tubes.

7.3. Flow meter

- 7.3.1. Use a flow meter certified traceable to NIST standards.

7.4. Tube conditioning system (Model TC1, GERSTEL Inc, Baltimore, MD, or equivalent)

- 7.4.1. Tube effluent is directed to vent without passing through key parts of the sample flow path (e.g. the focusing trap).
- 7.4.2. Conditioning system must be leak-tight to prevent air ingress.
- 7.4.3. Conditioning system must allow precise and reproducible temperature selection ($\pm 5^\circ\text{C}$), at a temperature range at least as great as that of the thermal desorber.
- 7.4.4. Conditioning system must support inert gas flows in the range of 50 to 100 mL/min.

7.5. Brass, 1/4" Swagelok® -type fittings and PTFE ferrules

7.6. Uncoated aluminum foil

7.7. 2- μm Teflon® filter cartridge or short clean tubes containing loose plugs of clean glass wool

7.8. FEP tubing (FEP-MET-8 \times 6-TE-1, Norell Inc) and 1/4" Teflon tubing

8. Procedures

8.1. Tube labeling and identification

- 8.1.1. All sorbent tubes are pre-labeled by manufacturer with unique numbers.
- 8.1.2. Document the following information (some of which will need to be obtained from the manufacturer) for each tube: masses and/or bed lengths of sorbent(s), maximum allowable temperature, packing or repacking date, each date on which the tube was conditioned, date of use, and the date that the tube's SSV was tested (Section 10.4.1).
- 8.1.3. Check conditioning by obtaining chromatograms (SOP V4) to verify the absence of contaminating artifacts. Keep and store all such chromatograms together with the other written records for each tube.
 - 8.1.3.1. For new tubes, analyze a minimum of 10% of the tubes in each batch.

8.2. Conditioning and subsequent handling

8.2.1. Follow the TC1 Instrument Manual to condition tubes.

8.2.1.1. Purchased, preconditioned tubes should be conditioned for a minimum of 30 min. To do so, they should be brought to the temperatures given in Table 3, while a pure helium carrier gas is passed through them, at the flow rate given in Table 3.

8.2.1.2. If the tube history is unknown, it should be conditioned for a minimum of 2 h.

8.2.1.3. Up to 10 tubes may be conditioned at one time using the TC1.

8.2.2. Obtain post-conditioning chromatograms (SOP V4) for the required number of tubes (Section 10.7).

8.2.3. Once conditioned, seal the tubes in the storage container provided by the manufacturer. Never use greased fittings. Wrap the sealed tubes individually in uncoated aluminum foil, and place them in a clean, airtight, opaque container.

8.2.4. Add a package of clean sorbent material, e.g. activated charcoal or activated charcoal/silica gel mixture, to the container.

8.2.5. If tubes will not be used within 24 h of conditioning, store them in a refrigerator (organic solvent-free) at 4°C. See Section 5.4.1 concerning the use of tubes that have not yet returned to ambient temperature.

8.2.6. On second and subsequent uses, tubes will generally not require further conditioning. An exception to this is tubes exposed in their immediate prior use to high levels of VOCs; these should be reconditioned prior to continued usage.

8.3. Preparation for sample collection

8.3.1. At the monitoring location, keep the tubes in their storage and transportation container until they have reached ambient temperature.

8.3.2. Using clean gloves, remove the sample tubes from the storage container, remove their caps and attach them to the sampling lines. Use 6 mm ID FEP tubing for these connections. Connect two tubes in series using a Teflon tubing union, formed by bending a piece of ¼" Teflon tubing into a "U" shape and inserting each end into a short piece of 6-mm ID FEP tubing to allow a connection with the sorbent tube.

8.3.3. Uncap and immediately reseal the required number of field blank tubes (See Section 10.1.2). Place the field blank tube(s) back in the storage container.

8.4. Set sampling flow rates

8.4.1. The sampling train (Fig. 1) consists of the following: in-line particulate filter (Optional, Section 5.6), ozone scrubber (Optional, Section 5.3), sampling tubes (two in series), and a flow meter/pump combination (Section 7.2).

8.4.1.1. All connections coming into contact with the sample stream (all connections before the second tube) must be Teflon, to minimize offgassing. Connections after the sorbent tubes can be standard flexible Tygon tubing.

8.4.2. Collect samples over 1 h, with a sampling rate of 50 mL/min or 100 mL/min, depending upon whether 3 L or 6 L of air are to be sampled (Section 10.3).

8.4.3. Establish the approximate sampling flow rate using two "dummy" tubes of identical construction and packing as the sampling tube to be used. Set the flow rates of the pump using the flow meter, which is placed in line after the second tube. Record the flow rate as reached during this period.

8.4.4. Once the flow rate has been established with the "dummy" tubes in place, and has held stable for one minute, remove the "dummy" tubes and place the sampling tubes

- on the sampling train. Make final adjustments to the flow meter as quickly as possible, so as to avoid significant errors in the sample volume. Record the flow rate which is used for the sampling.
- 8.4.4.1. If multi-bed sorbent tubes are used, they must be oriented so that the air sample passes through a series of sorbents in order of increasing sorbent strength (i.e., weaker sorbent first). This is necessary to prevent contamination of the stronger adsorbent with less-volatile components.
- 8.5. Sample and recheck flow rates
 - 8.5.1. Sample over the specified sampling period (i.e. 1 h). Recheck all the sampling flow rates at the end of the sampling period, just before switching off the pump. Record the flow rate every 5 to 10 min during the sampling event.
 - 8.5.2. The flow rate measured at the end of sampling must be within 10% of that at the start of the sampling period, or the sample must be invalidated. Use the average value between the beginning and ending flow rates for all calculations.
 - 8.5.3. Make notes of all relevant monitoring parameters: locations, tube identification numbers, pump flow rates, dates, times, sampled volumes, ambient conditions, etc.
 - 8.6. Reseal the tubes
 - 8.6.1. Immediately remove the sampling tubes with clean gloves, put them back in the storage container, rewrap with uncoated aluminum foil, and place in a clean, opaque, airtight container. Do not store “loaded” sampling tubes in the same container as clean tubes.
 - 8.6.2. If tubes will not be analyzed during the same day, place the container in a clean (organic solvent-free), refrigerator (< 4°C) or freezer until they are to be analyzed.
 - 8.6.3. In all cases, conduct analysis within 30 d of sample collection.
 - 8.7. Shipping
 - 8.7.1. Ship all samples by the fastest available means (preferably overnight), and include sufficient amounts of “blue ice” (or equivalent) to keep cold. Avoid shipping that will result in arrival of the samples on days when laboratory personnel will not be available to receive samples.
 - 8.7.2. Include chain-of-custody forms with all sample shipments.
 - 8.8. Receipt of samples
 - 8.8.1. Note condition and temperature of sample containers.
 - 8.8.2. Log in samples such that analyses can be scheduled (Section 8.6.3).
 - 8.8.3. Store samples (Section 8.6.2).
 - 8.8.4. Sign and date chain-of-custody forms.

9. Data and Records Management

- 9.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
- 9.2. Manage all data according to SOP B5.
- 9.3. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes.
- 9.4. Overstrike all errors in writing with a single line. Initial and date all such corrections.

10. Quality Control and Quality Assurance

10.1. Blanks

10.1.1. Lab blanks must be identically prepared tubes, from the same batch, with similar histories and conditioned at the same time as the tubes used for sample collection. At least two blanks are required for each 10 tubes (a “batch”) used in a single sampling event (which may stretch over one or more days). If more than 10 samples are taken in a single event, they must be divided into two (or more, as appropriate) batches for the purpose of including blanks. Lab blanks must be stored in the lab in clean controlled conditions (Sections 8.2.4. and 8.2.5). They should be analyzed at the same time as the samples - one at the beginning and one at the end of the samples’ analytical sequence.

10.1.2. Field blanks are the same as lab blanks; however, they are transported to and from the monitoring site, and are uncapped and immediately resealed at the monitoring site. They do not actually have air pumped through them. One field blank tube is taken for every ten tubes included in a sampling event (one tube per batch), with the exception that no less than two field blanks may be included, however small the sampling event. Field blanks should be distributed evenly throughout the run when the tubes are analyzed.

10.1.2.1. If the profile/pattern of VOCs on the field blank matches the sampled tubes, and if the areas of peaks in the field blanks are >5% of those in the sampled tubes, pay careful attention to the method of sealing tubes and other storage procedures in future sampling events. If the areas of peaks of field blank are >10% those in the sampled tubes, then invalidate the sampled tube data.

10.2. Determination and validation of SSVs

10.2.1. If SSV information is not readily available for the analytes under test on the sorbent tube selected, or if the SSVs need validating, use the following field experiment, taken from Method TO-17.

10.2.1.1. The experiment should be carried out in the atmosphere to be monitored and, if possible, under worst-case conditions (i.e., highest natural humidity and highest typical VOC concentrations). Select a well-ventilated sampling location.

10.2.1.2. Link 2 sorbent tubes together in series. Repeat this five additional times, so that a total of 12 tubes (six pairs) are used.

10.2.1.2.1. Use Teflon tubing unions as described in Section 8.3.2.

10.2.1.3. Connect the sampling end of the backup tube to the exit end of the primary tube.

10.2.1.4. Connect the tube pairs to individual calibrated monitoring pumps, and simultaneously sample at least 3 different air volumes.

10.2.1.4.1. Place the sampling ends of all the tube pairs close together, as much as possible, to ensure that all tubes are sampling the same atmosphere.

10.2.1.4.2. Use pump flow rates between 10 and 200 mL/min.

10.2.1.4.3. Obtain 2 replicates at each air sample volume.

10.2.1.5. Analyze both the primary and backup tubes of each pair by GC/MS (SOP V4).

10.2.1.6. If more than 5% of one or more of the target analytes is observed on any of the backup tubes, breakthrough is shown to have occurred at that sample volume.

- 10.2.1.7. If the amount of analyte found on the backup tube at a particular sampling volume is in the range of 5-10% (i.e. the sampling volume is at or very close to the actual BV), set the SSV as two thirds (~66%) of that volume.
 - 10.2.1.8. If the amount of analyte found on the backup tube at a particular sampling volume is greater than 10% (i.e. the sampling volume is considerably beyond the actual BV), set the SSV as the next lowest sampled volume (provided that volume does not display breakthrough).
- 10.3. Initial testing of each sampling site
- 10.3.1. The first time a sampling location is tested, such as during an initial site characterization, is considered to be an “uncharacterized atmosphere” (using terminology from Method TO-17). Conduct a scaled-down, site-specific (as opposed to compound-specific) version of the experiment detailed in Step 10.2.
 - 10.3.1.1. In these cases, collect distributed volume tube pairs (e.g. 1- and 4-L samples) in parallel (i.e. simultaneously from one sampling location, per Section 10.2.1.4.1).
 - 10.3.1.2. Ideally, the quantity of material collected (after quantitation by GC/MS) should scale linearly with sample volume on a plot forced through the origin.
 - 10.3.1.2.1. If this condition is met (i.e. if the R^2 value for the line is ≥ 0.98), use the larger sample volume (4 L) in future equivalent (i.e. within the same season) sampling events.
 - 10.3.1.2.2. If the R^2 value for the line is < 0.98 , and the amount recovered in the 4-L sample is less than 4 times that in the 1-L sample, use the smaller (1-L) volume in future sampling events.
 - 10.3.1.2.3. If the R^2 value for the line is < 0.9 , and/or the amount recovered in the 4-L sample is more than 5 times that in the 1-L sample, repeat the test, as a sampling error has occurred.
 - 10.3.1.3. Backup tubes should be used to investigate situations in which distributed volume pairs do not agree within acceptable tolerance. This will rule out the possibility of experimental error, and validate the decision to use the smaller sampling volume at a given site.
- 10.4. Instrument calibration and performance criteria
- 10.4.1. Calibrate pumps according to the manufacturer’s instructions.
 - 10.4.2. Calibrate flow meters annually (or as specified in the QAPP) according to SOP A9.
 - 10.4.3. Calibrations conducted at the monitoring location, immediately before sampling begins, are preferred. Alternatively, conduct calibration in a clean environment before the tubes and pumps are transported to the monitoring site.
 - 10.4.4. Calibrate pumps with the type of sorbent tube to which they will be connected during the actual sampling.
 - 10.4.5. Any pump that “fails” Step 8.5.2 (i.e. does not maintain a flow rate for 1 h within 10% of its initial set rate) more than once will be repaired and recalibrated.
 - 10.4.6. Review records of the pump flow rate delivered against the pump flow set rate, stroke rate or pressure at least once per 3 mos. If the performance of any pump has been found to have changed significantly over that time (for example, the flow rate measured at the end of sampling doesn’t agree within 10% of the start of sampling, or if completely different pump settings are required to deliver the same pump flow rate), have the pump serviced by the manufacturer or their approved agent.

10.4.7. Certify all flow meters traceable to National Institute of Standards and Technology (NIST) standards.

10.5. Performance criteria for sorbent tubes sampling of air

10.5.1. Precision of duplicate pairs

10.5.1.1. The measure of analytical precision used for this SOP is the absolute value of relative difference between two identical samples.

10.5.1.2. The identical samples must have the same flow rate over the same time period from a common inlet.

10.5.1.3. Calculate the analytical precision as follows:

$$\text{Precision} = \frac{[X_1 - X_2]}{X} \times 100$$

Where:

X_1 = One measurement value (e.g., for a defined sample volume of 1L)

X_2 = Duplicated measurement value

X = Average of X_1 and X_2

10.5.1.4. Agreement within 25% for distributed volume pairs should be met.

10.6. Audit accuracy

10.6.1. Prepare gaseous audit standards using any of the four methods described in Section 9.1 of SOP V6. Concentrations of analytes in the audit standard should be such that the amount trapped on the sorbent tube, given a 4-L sample volume, is approximately in the middle of the concentration range used for the multipoint calibration curve.

10.6.2. Collect the audit standards onto an adsorbent tube with sampling train, following the procedures in this SOP. Collect duplicate samples of the audit standard.

10.6.3. Analyze the audit standard tubes by GC/MS (SOP V4).

10.6.4. The audit accuracy is the relative difference between the measurement result and the nominal concentration of the audit compounds:

$$\text{Audit Accuracy, \%} = \frac{(\text{Spiked} \cdot \text{Value} - \text{Observed} \cdot \text{Values})}{(\text{Spiked} \cdot \text{Value})} \times 100$$

10.6.4.1. Audit accuracy must be within 25%.

10.7. "Spot-checks" of sorbent tube conditioning

10.7.1. Initially (i.e. at the start of a project), analyze all tubes individually by GC/MS to verify their proper conditioning.

10.7.2. After the conditioning system has been proven reliable, spot-check a lower percentage of tubes, and certify the entire batch.

10.7.2.1. Check at least 10% of any given batch of tubes, where "batch" refers to the group of tubes conditioned in a given session.

10.7.2.2. Tubes in a batch which are to be spot-checked should have contained similar contaminant levels as a result of their most recent sample collection. If some tubes in a batch are known to have contained higher levels of contaminants than others, give preference to checking some (or all) of these.

11.**11. References**

- 11.1. ASTM D 6196-03. 2003. Standard Practice for Selection of Sorbents, Sampling, and Thermal Desorption Analysis Procedures for Volatile Organic Compounds in Air. ASTM International, West Conshohocken, PA.
- 11.2. EPA Method TO-17. 1999. Determination of Volatile Organic Compounds in Ambient Air Using Active Sampling onto Sorbent Tubes. In Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition (EPA/625/R-96/010b). Center for Environmental Research Information, Office of Research and Development, US EPA, Cincinnati, OH.
- 11.3. TC1 Tube Conditioner Manual. GERSTEL GmbH & Co.KG, Mülheim an der Ruhr, Germany.
- 11.4. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 11.5. SOP V4. 2006. Thermodesorption and GC/MS Analysis of VOCs Collected on Sorbent Tubes. Standard Operating Procedure V4. Purdue Ag Air Quality Lab.
- 11.6. SOP V6. 2006. Standard Operating Procedure For GC/MS Analysis of VOCs Collected in Sampling Canisters. Standard Operating Procedure V6. Purdue Ag Air Quality Lab.

Table 2. Analyte suitability of different common, commercially available sorbent matrices used in air sampling tubes.

Sample Tube Sorbent	Approx. Analyte Volatility Range	Specific Surface Area (m ² /g)	Example Analytes
CarbotrapC® CarbopackC® Amsorb® GCB2	n-C ₈ to n-C ₂₀	12	Alkyl benzenes and aliphatics
Tenax® TA	bp 100°C to 400°C n-C ₇ to n-C ₂₆	35	Aromatics (except benzene), Apolar components (bp>100°C) and less-volatile polar components (bp> 150°C)
Tenax GR	bp 100°C to 450°C n-C ₇ to n-C ₄₀	35	Alkyl benzenes, vapor-phase PAHs and PCBs and as above for Tenax TA.
Carbotrap® CarbopackB® Amsorb® GCB1	(n-C ₄) n-C ₅ to n-C ₁₄	100	Wide range of VOCs, including ketones, alcohols, and aldehydes (bp>75°C) and all apolar compounds within the volatility range specified.
Chromosorb® 102	bp 50°C to 200°C	350	Wide range of VOCs, including oxygenated compounds and haloforms less volatile than methylene chloride.
Chromosorb 106	bp 50°C to 200°C	750	Wide range of VOCs, including hydrocarbons from n-C ₅ to n-C ₁₂ . Also good for volatile oxygenated compounds.
Porapak Q	bp 50°C - 200°C n-C ₅ to n-C ₁₂	550	Wide range of VOCs, including oxygenated compounds.
Porapak N	bp 50°C to 150°C n-C ₅ to n-C ₈	300	Specifically selected for volatile nitriles: acrylonitrile, acetonitrile and propionitrile. Good for pyridine, volatile alcohols from EtOH, and for methylethylketone (MEK).
Spherocarb	-30°C to 150°C C ₃ to n-C ₈	1200	Good for very volatile compounds such as ethylene oxide, and dichloromethane. Also good for polar VOCs, e.g. MeOH EtOH and acetone.
Carbosieve SIII® Carboxen 1000® Anasorb® CMS	-60°C to 80°C	800	Good for ultra-volatile compounds such as low-molecular-weight hydrocarbons, volatile haloforms and freons.

Table 3. Appropriate conditioning parameters to be used for sampling tubes containing various sorbent matrices.

Sample Tube Sorbent	Hydrophobic (?)	Conditioning parameters	
		Temperature (°C)	Gas flow (mL/min)
CarbotrapC® CarbopackC® Amsorb® GCB2	Yes	350	100
Tenax® TA	Yes	330	100
Tenax GR	Yes	330	100
Carbotrap® CarbopackB® Amsorb® GCB1	Yes	350	100
Chromosorb® 102	Yes	250	100
Chromosorb 106	Yes	250	100
Porapak Q	Yes	250	100
Porapak N	No	180	100
Spherocarb	Yes	400	100
Carbosieve SIII® Varbpxem 1000® Anasorb® CMS	No	350	100
Zeolite Molecular Sieve 13X	No	330	100
Tenaz / CB: comb. Tube Type 1	Yes	330	100
Carb B / CMS: comb. Tube Type 2	No	350	100
Carb 300 type: comb. Tube Type 3	No	350	100

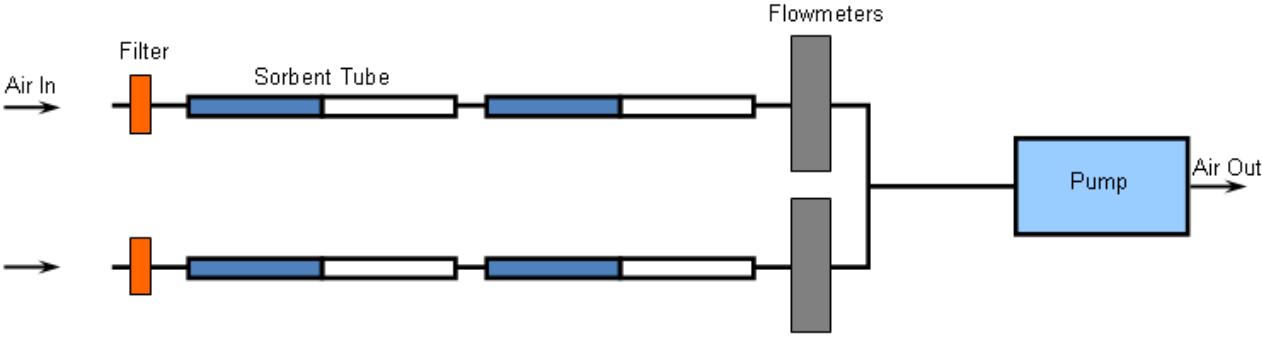


Figure 1. Diagram of the sampling train, as set up for duplicate samples at one location.

VOC SAMPLING USING CANISTERS
Standard Operating Procedure (SOP) V2

VOC SAMPLING USING CANISTERS
Standard Operating Procedure (SOP) V2

Prepared by

Juan Carlos Ramirez, Bill W. Bogan, and Hua Xu

Reviewed by

Albert J. Heber

Effective Date: November 13, 2007

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability.....	3
2. Summary of Method.....	3
3. Definitions.....	4
4. Health and Safety.....	4
5. Cautions.....	4
6. Interferences.....	4
7. Personnel Qualifications.....	5
8. Equipment and Supplies.....	5
9. Procedure.....	6
10. Data and Records Management.....	9
11. Quality Control and Quality Assurance.....	9
12. References.....	10
Appendix A. VOC Canister Field Test Data Sheet.....	11

1. Scope and Applicability

- 1.1. A method is needed to sample volatile organic compound (VOC) emissions from barns and other buildings at livestock facilities.
- 1.2. This procedure describes the subatmospheric pressure sampling of VOCs using canisters. VOC analysis is described in SOP V6, while the procedures necessary to ensure the cleanliness of the canisters themselves, as well as the other components of the sampling system, are described in SOP V7.
- 1.3. Time-integrated sampling is conducted in indoor ambient atmospheres at confined animal feeding operations (CAFOs). The typical sampling period, in accordance with USEPA Methods TO-14A and TO-15, and ASTM Method D5466-01, is 24 h.
- 1.4. The “whole air” sample is collected in stainless steel canisters with specially treated (passivated) interior surfaces (SilcoCan® canisters). Such canisters are rugged, proven and a convenient way of collecting air samples in the field. They can be cleaned and reconditioned (SOP V7) and reused for subsequent samples.
- 1.5. This procedure is applicable to specific VOCs that have been tested and determined to be stable when stored in subatmospheric pressure canisters. A list of applicable analytes, along with approximate detection limits, are provided in SOP V6. Analyte stability tests are described in detail in SOP V7.
- 1.6. When sampling under negative pressure (i.e. samples are drawn into the canister by the vacuum inside), the maximal volume which can be sampled is the volume of the canister. Pressurized systems (i.e. those incorporating bellows-type pumps), although not applicable under this SOP, can collect larger volumes.

2. Summary of Method

An ambient air sample is drawn into a conditioned canister through a sampling system (Fig. 1) that includes: a short sampling inlet, a 2- μm particulate filter, a spring washer, an interchangeable critical orifice to restrict the flow range, a Veriflo® SC423XL flow controller to maintain constant sampling flow when a time averaged sample is required, a vacuum gauge and a canister. A canister is a stainless steel vessel in which the internal surfaces have been specially passivated using a unique Siltek® passivation technology, which chemically bonds a fused silica layer to the metal inner surface of the canister. SilcoCan® canisters are suitable for the sampling of most VOC analytes, including polar and sulfur-containing compounds. The canister is evacuated and used to collect an air sample, using the internal vacuum as the driving force. A canister will hold a vacuum >28 in. Hg (>13.8 psi) for up to 30 d. The canisters are subjected to various quality assurance (QA) performance checks, including leak checks and certification of both the canister and the sampling train, both of which are presented in SOP V7. After collection, the canister valve is closed, a tag is attached to the canister, a chain-of-custody (COC) form is completed and the canister is transported to the lab for analysis (SOP V6).

3. Definitions

3.1. atm	atmosphere. 1 atm = 29.92 in Hg (14.96 psi)
3.2. CAFO	Confined Animal Feeding Operation
3.3. Certification	The process of demonstrating, using humid zero air and humid calibration gases, that the sampling system will not change the concentrations of sampled and stored gases (SOP V7).
3.4. COC	Chain of custody
3.5. GSS	Gas sampling system
3.6. NIST	National Institute of Standards and Technology
3.7. PAAQL	Purdue Agricultural Air Quality Laboratory
3.8. PTFE	Polytetrafluoroethylene
3.9. "Practice" Canister	A canister used to calibrate the mass flow controller
3.10. QAPP	Quality Assurance Project Plan
3.11. sccm	standard cubic centimeters per minute
3.12. VOC	Volatile organic compound(s)

4. Health and Safety

- 4.1. Each person needs personal protection equipment according to the place where the work is being conducted.
- 4.2. Release or evacuate air from a canister to a fume hood or to outside.
- 4.3. Wear safety glasses when handling canisters; some suppliers send canisters pressurized.

5. Cautions

- 5.1. Avoid sources of impurities, such as the following:
 - 5.1.1. Organic compounds outgassing from the system components ahead of the canister
 - 5.1.2. Solvent vapors in any location other than the one which is to be sampled
- 5.2. Canister valves must be closed at the exact end of sampling period. If not, the canister pressure will be lower than the pressure required by the flow controller. The flow in the last portion of the sampling cycle will not be at the same value that the initial flow.
- 5.3. For reasons specified in Section 6.1, do not exceed 30 d storage of samples prior to analysis.

6. Interferences

- 6.1. The following loss mechanisms for analytes collected into sampling canisters are generally considered (USEPA Compendium Method TO-15) to be short-term, and could affect the stability of analytes over the periods relevant to this SOP (i.e. the 2 weeks required to assess analyte stability in Section 11.8 of SOP V7). In general, however, most VOCs can be recovered from canisters near their original concentrations after storing them up to 30 d. Do not exceed 30 d storage of samples prior to analysis.
 - 6.1.1. Reactive sites on the internal surface(s) of the canister and/or sampling train can catalyze loss of analytes. Use only passivated canisters.

- 6.1.2. Losses due to physical adsorption occur as equilibrium is reached between the condensed and gas phases of an analyte. These are generally considered to occur over minutes to hours, and may be more significant with low-humidity samples.
- 6.1.3. Conversely, if there is any water condensed in the canister, analyte losses can occur by dissolution, particularly if humid samples are pressurized, leading to condensation.
 - 6.1.3.1. Loss mechanisms based on aqueous hydrolysis also exist.
- 6.1.4. Chemical reactions between VOCs and other, more stable substances inside the canister are generally assumed to cause the gradual decrease of concentration over time (i.e., long-term losses over days to weeks).
- 6.1.5. Biodegradation of analytes can also occur over time.
- 6.2. Never use non-chromatographic grade stainless steel tubing, non-PTFE thread sealants, or flow controllers with Buna-N rubber components.
- 6.3. Dust particles can obstruct the orifice, cause valve leaks, and possibly damage the valve. Use a particulate filter (Section 8.2.1, Figure 1).

7. Personnel Qualifications

- 7.1. Each person sampling VOCs using canisters must read and understand this SOP.

8. Equipment and Supplies

- 8.1. Equipment (Fig. 1)
 - 8.1.1. Stainless steel canister(s): Leak-free stainless steel pressure vessels of desired volume with valve and specially prepared (passivated) interior surface. For the purposes of this SOP, SilcoCan[®] canisters (Cat. # 24142, Restek, Bellefonte, PA) or equivalent) will be used.
 - 8.1.2. Passive air sampling kit (Restek Cat. #24160), which includes the following:
 - 8.1.2.1. Stainless steel sampling tube (¼" ID).
 - 8.1.2.2. 2-µm frit filter
 - 8.1.2.3. Spring washer
 - 8.1.2.4. Critical orifice (0.0012-in. dia.)
 - 8.1.2.5. Stainless steel vacuum/pressure gauge capable of measuring a vacuum of 0–30 in. Hg (0–14.7 psi) and a pressure of 0–61 in Hg (0–30 psi) in the sampling system. All wetted surfaces are passivated stainless steel.
 - 8.1.2.6. Mass flow controller (Veriflo™ Model SC423XL, Restek Corp., Bellefonte, PA, or equivalent). Capable of maintaining a constant flow of 2-4 sccm (± 10%) over sampling periods up to 24 h and under conditions of changing temperature (20 – 40°C) and humidity.
 - 8.1.2.6.1. The specific part number for the MFC is dependent on the desired flow range. Flow ranges outside 2-4 sccm would require a different MFC.
 - 8.1.3. Electronic time meter/timer/stopwatch
 - 8.1.4. Certified mass flow meter (to verify sampling train flow controller) (Allicat Scientific, M-50 sccm, air)
 - 8.1.5. 9/16" wrench
 - 8.1.6. 3-mm Hex (Allen) wrench (for adjusting the Veriflow Flow controller).

8.2. Supplies

- 8.2.1. Particulate matter filter: 2-micron sintered stainless steel in-line filter
- 8.2.2. Chain-of-custody forms (original and two copies each-field and lab copies).

9. Procedure

9.1. Sampling train preparation

- 9.1.1. Sampling train cleaning, assembly, leak testing and certification as per SOP V7.
- 9.1.2. Check the sampler flow controller in the lab, as follows:
 - 9.1.2.1. Install a “practice” canister (evacuated to the same pressure as the actual sampling canister) to the sampling train to test the flow controller.
 - 9.1.2.2. Connect 7- μ m filter in front of digital flowmeter with 1/4" Teflon tubing; connect the other end of flow meter to the inlet of sampling kit using a 1/4" Teflon union.
 - 9.1.2.3. Open the canister valve to apply vacuum.
 - 9.1.2.4. Remove the protective cap from the back of the Veriflo™ Flow Controller SC423XL body. With a 3-mm hex (Allen) wrench, adjust the piston gap screw to achieve the desired flow rate (2.8 – 3.5 ml/min). Allow the flow to equilibrate for several minutes between adjustments.
 - 9.1.2.5. Replace the protective cap onto the back of the Veriflo™ Flow Controller body.
 - 9.1.2.6. Turn off the canister valve
- 9.1.3. Disconnect the "practice" canister and certified mass flow meter, and pack canister(s) and sampling train(s) for transport to field.

9.2. Sampling procedure

- 9.2.1. Open the canister and sampling train boxes and verify that the chain-of-custody (COC) forms (original and field copy) are included.
- 9.2.2. Read the pressure of the canister from the vacuum gauge on the sampling kit.
 - 9.2.2.1. Confirm that the canister valve is closed.
 - 9.2.2.2. Remove the plug nut that is at the open side of the canister valve.
 - 9.2.2.3. Attach a pressure gauge to the canister.
 - 9.2.2.4. Open and close valve quickly (a few seconds).
 - 9.2.2.5. Read the vacuum on the gauge.
 - 9.2.2.6. Record the gauge reading on the “Initial Vacuum” column on the canister chain-of-custody form.
 - 9.2.2.6.1. The vacuum reading should be 0.05 in. Hg (0.025 psi). If it is not, record the pressure, make a note in the COC, and return the canister to the lab. Keep the COC field copy in the field canister file.
 - 9.2.2.7. Verify that the canister valve is closed and remove the gauge.
 - 9.2.2.8. Replace the plug nut to the canister valve.
 - 9.2.2.9. As an alternate procedure, install the sampling train and record the pressure indicated by the sampling train pressure gauge.
- 9.2.3. Position the sampling inlet
 - 9.2.3.1. If the air is dusty (i.e. in highly trafficked areas), the inlet must be positioned at least 2 m above the ground (EPA Methods TO-14A and TO-15) to minimize the entrance of dust particles.
 - 9.2.3.2. If the ambient air is not dusty, install the inlet at least 12 in. above ground.

- 9.2.3.3. For barns and livestock buildings, sampling will generally be conducted at the inlet(s) of exhaust fan(s)
- 9.2.4. Attach a clean, certified (SOP V7) canister to the system.
- 9.2.5. Open the canister valve, to allow air sample into the canister, and the vacuum pressure gauge.
- 9.2.6. Record the pressure/vacuum in the canister (as indicated by sampler vacuum/pressure gauge) on the "VOC Canister Field Test Data Sheet", and then close the vacuum pressure gauge. Also record time of day and current ambient and interior temperatures. Reset the max/min thermometer to capture the maximum and minimum temperatures during the 24-h sampling period.
- 9.2.7. Set the electronic timer, and program it to indicate the end of the sampling period.
- 9.2.8. At the conclusion of the sampling period, briefly open the vacuum/pressure gauge valve on the sampler, and record the pressure/vacuum on the "VOC Canister Field Test Data Sheet". Close the valve. Record the pressure, the time of day, and elapsed sampling time. Maximum and minimum temperatures during the sampling period can be retrieved from AirDAC.
 - 9.2.8.1. There are four possible scenarios for final canister pressure:
 - 9.2.8.1.1. Ideally, there will be a vacuum of 7"-4" Hg remaining in the canister.
 - 9.2.8.1.2. If >7" Hg vacuum remains, less sample was collected than initially anticipated. The sample will be valid, but the detection limit may be higher than expected. It may be necessary to pressurize the canister (SOP V6) prior to analysis, which will dilute the sample. A dilution factor will then be required to determine final target compound concentrations.
 - 9.2.8.1.3. A vacuum <4" Hg indicates the sample might be skewed toward the initial part of the sampling period. This assumption usually is valid because the flow rate through the flow controller will fall once the vacuum falls below 5" Hg, when the change in pressure across the flow controller diaphragm becomes too small to maintain a constant flow. The sample might still be usable, because sample was collected over the entire interval.
 - 9.2.8.1.4. If the ending vacuum is <1" Hg, the sample is invalid, because it will be impossible to tell when the sample flow stopped.
- 9.2.9. Disconnect the sampling train from the canister.
- 9.2.10. Complete the identification tag on the canister, which must include the canister serial number, sample number, location, and date.
- 9.3. Shipment of samples
 - 9.3.1. When a canister has been filled with a sample, include the canister information in the sample log notebook. The information is also included in the COC form.
 - 9.3.2. Include the original COC in the box with the canister, and keep the field copy in the field canister file in the trailer.
 - 9.3.3. Pack canister so that the valve is protected from disturbances that might cause leaks.
 - 9.3.4. Ship the canisters to the laboratory.

9.4. Calculations

9.4.1. Sampling flow calculation

9.4.1.1. To obtain the sampling flow to fill a canister use the following equation:

$$Q = \frac{P \times V}{T \times 60}$$

Where:

Q = flow rate, mL/min

P = final canister pressure (absolute fraction), given a “full canister” pressure of 1 atm and a “fully evacuated canister” pressure of 0 atm

V = volume of canister, mL

T = sample period, h

For example, if a 6-L canister is being used to sample for a 24-h period, and a final pressure of 7 in. Hg (3.44 psi), required by the Veriflo™ flow controller to maintain constant flow, is desired, the flow rate can be calculated by:

Final pressure (vacuum): 7 in Hg = 3.44 psi vacuum = 0.234 atm

Final pressure absolute = 1 atm – 0.234 atm = 0.766 (unitless)

$$Q = \frac{0.766 \times 6000}{24 \times 60} = 3.19 \text{ mL/min}$$

9.4.2. Sampling volume calculation (based on pressure)

9.4.2.1. Sample volume collected can be determined as follows:

$$\text{Sample volume} = \frac{(\text{Initial pressure}) - (\text{Final pressure})}{\text{Atmospheric reference pressure}} \times \text{Canister volume}$$

For example: A sample is collected in a 6-L canister. The initial pressure gauge reading when the canister left the lab was 29" Hg vacuum, and the final pressure gauge reading at the conclusion of sampling was 7" Hg vacuum.

$$\text{Sample volume} = [(29" \text{ Hg} - 7" \text{ Hg}) / 29" \text{ Hg}] \times 6 \text{ L} = 4.55 \text{ L collected.}$$

9.5. Troubleshooting

9.5.1. If there is no flow through the sampling train:

9.5.1.1. Check the filter to verify that it is not clogged with particles.

9.5.1.1.1. Clean the filter if necessary.

9.5.1.1.1.1. Disassemble the filter.

9.5.1.1.1.2. Remove the larger particles from the frit by blowing particle-free nitrogen through the frit from the outlet surface toward the inlet surface.

- 9.5.1.1.1.3. After larger particles are removed, sonicate or rinse filter parts in methanol.
- 9.5.1.1.1.4. Bake the parts in an oven at 130°C to remove any residual organic vapors.
- 9.5.1.2. Check the orifice to verify it is not obstructed with particles. This is more critical when using very small orifices.

10. Data and Records Management

- 10.1. Maintain all lab records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
- 10.2. Manage all data according to SOP B5.
- 10.3. Document all data and information on field (or lab) data sheets, and within site (or lab) logbooks with permanent ink, or in electronic field notes.
- 10.4. Maintain a permanent log for each individual canister and sampling system, which must include, at minimum, the following information (SOP V7):
 - 10.4.1. Dates and parameters of all cleanings (i.e. temperature applied, number of cycles required to achieve certification)
 - 10.4.2. Certification dates
 - 10.4.3. Data (i.e. chromatograms) on which each certification of cleanliness is based
 - 10.4.4. Dates and results of all leak tests
 - 10.4.5. Dates and results of all analyte stability checks
 - 10.4.6. Dates and locations of all sampling events, as well as analytical results obtained from each sample
 - 10.4.7. (For sampling systems) Dates and results of all dynamic calibration tests
- 10.5. Sample tracking
 - 10.5.1. Record each sample collected by filling out all information required on the "VOC Canister Field Test Data Sheet".
 - 10.5.2. Canister pressure is recorded on site, and checked upon arrival in the lab to ensure canister is airtight during transport. Samples are invalidated if leaks are detected.
- 10.6. Overstrike all errors in writing with a single line. Initial and date all corrections on all forms.

11. Quality Control and Quality Assurance

- 11.1. Calibrate and leak-check all Veriflo™ flow controllers annually (SOP V7).
- 11.2. Calibrate all mass flow meters at the beginning of the project using a calibrated bubble meter (SOP A9).
- 11.3. Measure and record canister pressure at the following times:
 - 11.3.1. In the lab, before it is sent to the field. Pressure should be 0.05 mm Hg (0.025 psi).
 - 11.3.2. In the field before sampling.
 - 11.3.2.1. If canister pressure is higher than 0.05 mm Hg, make a note in the COC, along with the temperature, and return the canister to the lab.

- 11.3.2.2. If canister pressure is lower than 0.05 mm Hg, this indicates that the canister did not remain airtight during transport. If any leaks are determined to have occurred during shipping, do not use the canister.
- 11.3.3. After sampling, vacuum should be within 10% of 7 in Hg (3.44 psi). If vacuum is higher or lower than this range, the differential pressure through the flow controller was not correct throughout the sampling cycle, and the canister must be invalidated.
- 11.3.4. At the lab upon receipt, vacuum should be the same value reported in the field. If a leak occurred during transport to the lab, the sample must be invalidated.
- 11.4. Duplicate samples may occasionally be required to assess the reproducibility of this method. The frequency of replication will be determined by the QAPP for a particular project.

12.

12. References

- 12.1. ASTM Method D5466-01. 2006. Standard Test Method for Determination of Volatile Organic Chemicals in Atmospheres (Canister Sampling Methodology). ASTM International, West Conshohocken, PA.
- 12.2. Method TO-14A. 1999. Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS). In Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition (EPA/625/R-96/010b). USEPA, Center for Environmental Research Information, Office of Research and Development, Cincinnati, OH.
- 12.3. Method TO-15. 1999. Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS). In Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition (EPA/625/R-96/010b). USEPA, Center for Environmental Research Information, Office of Research and Development, Cincinnati, OH.
- 12.4. SOP A9. 2006. Measurement of Airflow Rate or Calibration of Air Sampling Instruments with Gilibrator 2 Calibration Systems. Standard Operating Procedure A9. Purdue Ag Air Quality Lab.
- 12.5. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 12.6. SOP G1. 2006. Gas Sampling System. Standard Operating Procedure G1. Purdue Ag Air Quality Lab.
- 12.7. SOP V6. 2006. GC/MS Analysis of Phenols and Volatile Fatty Acids (VFAs) Collected in Sampling Canisters. Standard Operating Procedure V6. Purdue Ag Air Quality Lab.
- 12.8. SOP V7. 2006. VOC Cleaning, Certification and Pre-Sampling Preparation of Sampling Canisters. Standard Operating Procedure V7. Purdue Ag Air Quality Lab.
- 12.9. RESTEK Canister Guide. 2002. A Guide to Passive Air Sampling Using Canisters. Catalog 59977B. RESTEK Corporation, Bellefonte, PA. Online at <http://www.restek.com/restek/images/external/59977A.pdf>. Accessed 3/20/06.

Appendix A.

VOC Canister Field Test Data Sheet

A. GENERAL INFORMATION

SITE LOCATION: _____

SITE ADDRESS: _____

SHIPPING ADDRESS: _____

SAMPLING DATE: _____ SHIPPING DATE: _____

CANISTER SERIAL NO.: _____ OPERATOR: _____

CANISTER LEAK: _____

CHECK DATE: _____

B. SAMPLING INFORMATION

	TEMPERATURE				PRESSURE
	INTERIOR	AMBIENT	MAXIMUM	MINIMUM	CANISTER PRESSURE
START					
STOP					

	SAMPLING TIMES			FLOW RATES	
	LOCAL TIME	ELAPSED TIME METER READING	MANIFOLD FLOW RATE	CANISTER FLOW RATE	FLOW CONTROLLER READOUT
START					
STOP					

SAMPLING SYSTEM CERTIFICATION DATE: _____

QUARTERLY RECERTIFICATION DATE: _____

Signature and Title

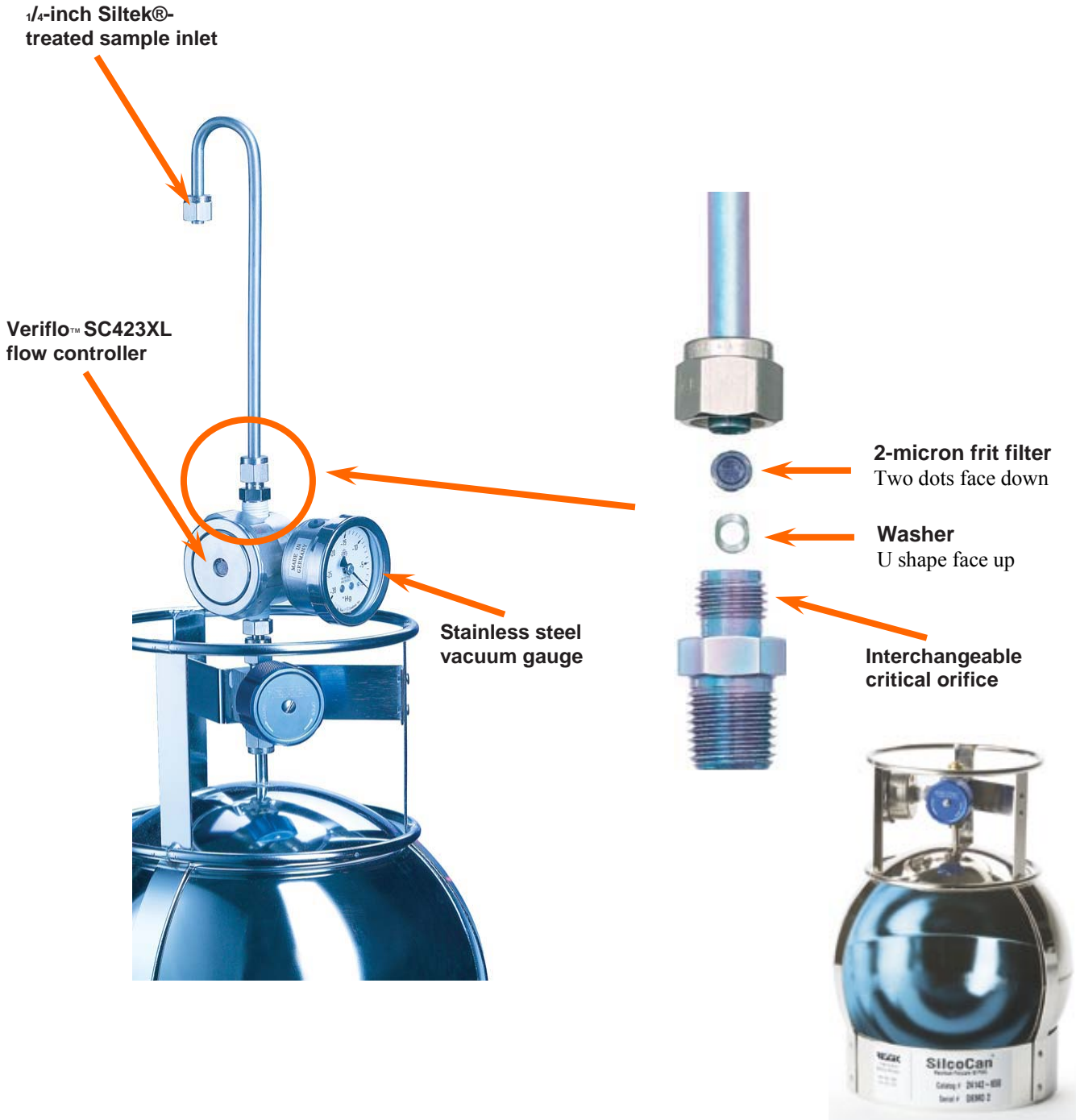


Figure 1. The negative-pressure passive sampling system used to sample air into canisters. Canister itself is shown at lower right.

**COLLECTION OF AMINES IN AIR SAMPLES THROUGH
USE OF SULFURIC ACID-CONTAINING BUBBLERS
Standard Operating Procedure (SOP) V3**

**COLLECTION OF AMINES IN AIR SAMPLES THROUGH
USE OF SULFURIC ACID-CONTAINING BUBBLERS
Standard Operating Procedure (SOP) V3**

Prepared by

Frank M. Mitloehner (Univ. of California-Davis) and Bill W. Bogan

Reviewed by

Congna Li and Albert J. Heber

Effective Date: November 13, 2007

**PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)
Agricultural and Biological Engineering, Purdue University
West Lafayette, IN 47907**

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions	3
4. Health and Safety	4
5. Interferences	4
6. Personnel Qualifications	4
7. Equipment and Supplies	4
8. Procedures.....	5
9. Data and Records Management.....	7
10. Quality Control and Quality Assurance.....	8
11. References	9

1. Scope and Applicability

- 1.1. Emissions of non-methane hydrocarbons or total VOC are regulated under the Clean Air Act and regulators are therefore interested in the VOC emissions rates from livestock production.
- 1.2. Amines released from livestock manure contribute to the total mass of NMHC.
- 1.3. Previous research has shown that amines in livestock air are efficiently sampled using sulfuric acid impingers or bubblers, and analyzed using ion chromatography.
- 1.4. Sampling periods are typically 120 min (at 1.0 L/min), but can be longer if the flow rate is lower, and if the pump can maintain this constant rate over the longer period.
 - 1.4.1. The goal is to sample 120 L of air, although this must be reduced if the concentrations of amines and/or ammonia are high enough to exceed the capacity of the trapping solution.
 - 1.4.1.1. This is indicated if the concentrations of amines (determined according to SOP V5) are equal in the first and second bubblers in the sampling train.
 - 1.4.2. Do not exceed a flow rate of 1.0 L/min.
- 1.5. Samples can be stored for up to 14 days prior to analysis.
- 1.6. The amines that can be captured from air with this method include primary amines (methylamine, butylamine) and secondary amines (dimethylamine, dibutylamine). A more complete list of target amines, along with approximate detection limits, is provided in SOP V5.

2. Summary of Method

The method is based on the South Coast Air Quality Management District (SCAQMD) Method 207.1 and the American Public Health Association (APHA) Method 124, "Tentative Method of Analysis for Primary and Secondary Amines in the Atmosphere (Ninhydrin Method)". A known volume of air is drawn from barn exhaust air through a series of bubblers, which contain 0.1 N sulfuric acid (H_2SO_4) solution. Upon reacting with the H_2SO_4 , the amines in the air stream are converted to their sulfate salts. For most amines, these salts are less volatile and more stable (e.g. more resistant to oxidation and chemical decomposition) than the free amine. The collected sample is then diluted for subsequent analysis (SOP V5).

3. Definitions

- 3.1. APHA American Public Health Association
- 3.2. Batch All bubbler trains to be used during a particular sampling event (which may extend over more than one day), but not to exceed ten. If more than ten trains will be used in an event, they must be grouped into more than one batch for the purposes of including blanks (Section 10.1).
- 3.3. HDPE High-density polyethylene
- 3.4. NMHC Non-methane hydrocarbons
- 3.5. SCAQMD South Coast Air Quality Management District

4. Health and Safety

- 4.1. Each chemical compound should be treated as a potential health hazard. Several aliphatic amines are highly flammable, and can cause severe eye damage; they are also capable of being absorbed through the skin. Some aromatic amines (e.g. certain toluidine isomers) are suspected carcinogens.
- 4.2. Be careful when working with H_2SO_4 to make the trapping solution.
 - 4.2.1. Wear protective clothing (gloves, lab coats, protective eyewear).
 - 4.2.2. Work in a properly-ventilated chemical fume hood.
 - 4.2.3. Always add H_2SO_4 to isopropanol, rather than isopropanol to H_2SO_4 .

5. Interferences

- 5.1. Ammonia will also be captured by this method. It is possible that, in sampling areas with very high ammonia levels, this could interfere, either by consuming the reactive capacity of the sampler, or by interfering with the subsequent IC analysis.
 - 5.1.1. Ammonia levels will also affect the post-sampling handling procedure for the bubblers. If ammonia concentration is high (above approximately 270 ppm, based on a 120-L sample volume), use 0.1 N H_2SO_4 , instead of distilled water, to rinse the bubblers (Step 7.4.3). Analyte losses (i.e. incomplete recovery) could occur otherwise. Consult ammonia monitoring data for the site location (SOP G4 or SOP G10) beforehand to make this determination.
- 5.2. When analyzing for volatile organics, samples can be contaminated (and/or cross-contaminated) by diffusion of volatile organics through the sample container septum during shipment and storage. Although it is unlikely that this will interfere with analysis of amines by this method, trip and method blanks prepared from dilute H_2SO_4 and carried through sampling and subsequent storage and handling, must serve as a check on such contamination. See Section 11.1 for further discussion of method and trip blanks.

6. Personnel Qualifications

- 6.1. Personnel should be trained in the use of all applicable instrumentation, in appropriate sample-handling procedures, and in appropriate safety procedures, before performing the activities in this SOP.
- 6.2. Each analyst must read and understand applicable equipment manuals and this SOP before performing these activities.

7. Equipment and Supplies

- 7.1. Amine trapping solution (0.1 N H_2SO_4): Add 5 mL of concentrated (98%) H_2SO_4 to 1.0 L of MilliQ water.
- 7.2. 30-mL midget bubbler with sintered glass filter on inlet (Part # 7532-20, Ace Glass, Vineland, NJ) or equivalent.

- 7.3. Flexible non-outgassing tubing: 7-mm ID flexible FEP tubing (Cat. # FEP-MET-9 × 7-TE, Norell Inc., Landisville, NJ), 6-mm ID FEP tubing (Cat. # FEP-MET-8 × 6-TE, Norell Inc., Landisville, NJ) or equivalent
- 7.4. Glass fiber filters: Binder-free, 47-mm diameter, 1- μ m pore size (VWR Catalog #28150-976, or equivalent)
 - 7.4.1. Inline filter cartridge capable of holding 47-mm filter discs
- 7.5. Teflon endcaps (or equivalent) for capping bubblers during transport: These can be made by sealing one end of a short length of 7-mm ID FEP tubing with a heat gun.
- 7.6. Gerstel Model GS gas sampler
- 7.7. In-line water/vapor trap (Cat #225-22-01, SKC, or equivalent)
- 7.8. Flow meter: Must control airflow between 1 and 2 L/min, to within $\pm 2\%$.
- 7.9. Glass fiber membrane filters
- 7.10. Filter-holding assembly: Borosilicate glass or Teflon. Gaskets and/or supports (if present) may be of silicon rubber, Teflon, or Viton.
- 7.11. Silica gel: Indicator type, mesh size 6-16
- 7.12. Wash bottles:
 - 7.12.1. One polyethylene wash bottle containing deionized water
 - 7.12.2. One polyethylene wash bottle containing reagent-grade acetone to rinse and speed the air drying of the water-rinsed components.
 - 7.12.2.1. Do not store acetone in plastic bottles; keep it in a glass container and transfer it to the plastic wash bottle when needed.
- 7.13. Graduated cylinders: Glass or high-density polyethylene (HDPE) graduated cylinders, 20- to 25-mL capacity
- 7.14. Sample bottles: Pre-cleaned (TraceClean®) amber borosilicate glass with fluoropolymer resin-lined solid-top caps. 40-mL capacity. VWR Catalog #15900-030 (or equivalent).
- 7.15. Parafilm®, or equivalent
- 7.16. Aqueous amine calibration (standard) solution (SOP V5) for field spiking – Use the highest concentration standard from the five-point set of standards.
- 7.17. Transfer pipette capable of delivering 5 mL volume.

8. Procedures

- 8.1. Bubbler preparation
 - 8.1.1. Place 15 ml of trapping solution in each bubbler to be used on a particular sampling event (which may consist of one or more days). Note that each sample collected will require two full bubblers and one empty bubbler. Include a minimum of one method blank (Section 10.1.1), one trip blank (Section 10.1.2), and one train for field spiking (Section 10.1.4) with each batch of ten sampling trains.
 - 8.1.2. Cap the bubblers for transport, and place them in a suitable (cushioned and sealed) container.
- 8.2. Preparation for sample collection
 - 8.2.1. At the monitoring location, put on clean gloves, and assemble the sampling train in the following order (Fig. 1): filter assembly with glass-fiber filter, two bubblers with trapping solution, one empty bubbler, in-line vapor trap, GS sampler, flow meter.

- 8.2.2. Remove the bubblers from the container and assemble the sampling train. Keep the inlet of the first bubbler in the train capped until ready to connect to the sampling line (Step 8.3.2). Use FEP tubing to connect the train components that come into contact with the sample stream. Bend ¼" Teflon tubing to a proper shape and length to connect the first two bubblers (containing trapping solution). At each end of this ¼" Teflon tube, insert the ¼" tube into a short piece of 6 mm ID FEP tube, then insert that into a short piece of 7 mm ID FEP tubing. This is necessary to form good connections with the bubbler inlets and outlets. Use this same connection to connect to the inlet of the first bubbler. Components after the second trapping solution-containing bubbler can be connected with standard flexible Tygon tubing.
 - 8.2.3. Uncap and then reseal the required number of field blank bubblers (Section 10.1.1.1). Keep the field blank bubblers uncapped for a length of time comparable to length of time that the bubblers used to assemble the sampling train were uncapped. Process the field blanks according to Section 8.4.
- 8.3. Sample collection
- 8.3.1. Sampling periods are typically 120 min (at 1.0 L/min), but can be longer if the flow rate is lower, and if the pump/sampler can maintain this constant rate over the longer period.
 - 8.3.1.1. The goal is to sample 120 L of air, although this must be reduced if the concentrations of amines and/or ammonia are high enough to exceed the capacity of the trapping solution.
 - 8.3.2. Establish the approximate sampling flow rate (1.0 L/min) using a "dummy" bubbler train identical to that which will be used for sampling. Set the flow rate of the GS sampler using a mass flow meter, which is placed in line after the filter assembly (Section 8.2.1). Record the flow rate as reached during this period.
 - 8.3.3. Once the flow rate has been established with the "dummy" bubbler train in place, and has held stable for one minute, remove the "dummy" bubbler train and connect to the sampling bubbler train.
 - 8.3.4. Leak-test the sampling train using the leak test program of the GS sampler. The leak rate must not exceed 0.02 L/min through the flow meter. If it does, locate and fix leaks, or switch to another bubbler train.
 - 8.3.5. Restart the sampler, and make final adjustments to the flow controller as quickly as possible, so as to avoid significant errors in the sample volume. Record the flow rate which is used for the sampling.
 - 8.3.6. Sample for approximately 120 min. Record exact starting and stopping times for sampling.
 - 8.3.7. Recheck the sampling flow rate at the end of the sampling period, just before switching off the sampler. Record the flow rate at the end of the sampling period.
 - 8.3.8. The flow rate measured at the end of sampling must agree within 10% with that measured at the start of the sampling period, or the sample cannot be considered valid. The average value between the beginning and ending flow rates should be used for all calculations.
 - 8.3.9. Make notes of all relevant monitoring parameters including locations, sampler flow rates, dates, times, sampled volumes, ambient conditions etc.

8.4. Post-collection sample handling

- 8.4.1. Depending on air stream humidity, the volume of liquid in the bubblers may have changed during sampling. Determine the volume of liquid in each of trapping solution-containing bubblers by pouring their contents into individual clean graduated cylinders. Record the volume from each bubbler on the data sheet.
- 8.4.2. Pour the contents of each graduated cylinder into a separate 40-mL amber borosilicate glass bottle.
- 8.4.3. Use the deionized water wash bottle to rinse out all interior surfaces of the two trapping solution-containing bubblers, as well as their corresponding graduated cylinders. Add this rinse water to the respective bottles for the two bubblers.
 - 8.4.3.1. Limit the volume of rinse water so that the total volume of each bubbler plus its rinses is no more than 30 mL. This will allow 20 mL of rinse water to be used in the lab to transfer the sample to a 50-mL volumetric flask.
 - 8.4.3.2. In general, it is preferred not to rinse glassware with acetone to dry it. If, however, acetone must be used to dry the glassware, do not combine the acetone rinse with any sample. Discard the acetone rinses in a proper manner.
- 8.4.4. Tighten the cap on each bottle securely, place each bottle in a plastic bag, and record a sample number and identifying information on the bag with an indelible marker. Alternatively, use prepared labels, if these can be firmly affixed to the bags and will not come off or deteriorate during storage and/or shipping on ice.
- 8.4.5. Field spikes
 - 8.4.5.1. Open the first sample bubbler of the train designated as the field spike. Pipette 5 mL of the aqueous amine calibration standard into the bubbler.
 - 8.4.5.2. Process this bubbler only (not the second) through the steps in Sections 8.4.1 – 8.4.4.
- 8.4.6. Place the samples on ice in a suitable cooler, and transport to the lab for analysis.
 - 8.4.6.1. Do not freeze samples at any point.
 - 8.4.6.2. If samples must be shipped to the lab, do so by overnight shipping, in an ice-filled cooler. If shipping is not possible on the same day as sample collection, store the samples in a refrigerator (4°C) until shipping. Do not ship such that the samples will be held in transit longer than overnight (i.e. over a weekend or holiday), nor such that they arrive at the lab on a weekend or holiday.
- 8.4.7. Store the samples in a refrigerator (4°C) until analysis.
- 8.4.8. Analyze the samples (by ion chromatography as described in SOP V5) no more than 2 weeks after collection.

9. Data and Records Management

- 9.1. Maintain all lab records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
- 9.2. Manage all data according to SOP B5.
- 9.3. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes. Overstrike all errors in writing with a single line, and initial and date all such corrections.

10. Quality Control and Quality Assurance

10.1. Blanks and field spikes

10.1.1. Collect a minimum of one method blank with each batch of samples. This will be processed and analyzed (See SOP V5) with that sample batch.

10.1.1.1. Prepare method blanks (field blanks) as follows. Prepare bubblers containing trapping solution, and ship them to the sampling location. As sampling is being conducted, open them and transfer the trapping solution to a clean graduated cylinder (for volume determination – Step 8.4.1), and then to a pre-cleaned borosilicate glass bottle (both bubblers of trapping solution may be combined into one bottle). Keep method blanks uncapped and open for a period of time comparable to the period which the actual sampling bubblers are uncapped. Rinse the two bubblers from the method blank with deionized water (Section 8.4.3), and add these rinsates to the bottle. Label this bottle, pack it together with the rest of the samples from that batch, and return it to the lab.

10.1.2. Include a minimum of one trip blank each time a set of samples is processed and analyzed.

10.1.2.1. Trip blanks will consist of clean bubblers which are filled at the lab with trapping solution, sealed, transported to the sampling location (but not opened), and then returned to the lab with the sample batch.

10.1.3. Blanks (either method or trip blanks) must not contain any analyte at greater than the detection limit. Corrective action if this occurs is discussed in SOP V5.

10.1.4. Field spikes

10.1.4.1. Collect a minimum of one method blank with each batch of samples. This will be processed and analyzed (SOP V5) with that sample batch.

10.1.4.2. Procedures for conducting field spiking are given in Section 8.4.6.

10.2. Instrument calibration

10.2.1. GS sampler

10.2.1.1. Calibrate according to the manufacturer's instructions.

10.2.1.2. Calibration should preferably be conducted at the monitoring location immediately before sampling begins. Alternatively, calibrate in a clean room before the bubblers and sampler are transported to the monitoring site.

10.2.1.3. Calibrate samplers with the type of bubbler to which they will be connected during the actual sampling.

10.2.1.4. Repair and recalibrate any sampler that "fails" Step 8.3.5 (i.e. does not maintain a flow rate for 120 min within 10% of its initial set rate) more than once.

10.2.1.5. Review records of the sampler flow rate delivered against the pump flow set rate, stroke rate or pressure at least every three months. If the performance of any sampler is found to have changed significantly over that time (for example, if completely different settings are required to deliver the same flow rate), the sampler should be serviced by the manufacturer or their approved agent.

10.2.2. Flow meter

10.2.2.1. All flow meters will be certified traceable to NIST standards.

10.2.2.2. Calibrate all mass flow meters at the beginning of the project using a calibrated bubble meter (SOP A9).

11. References

- 11.1. Occupational Safety and Health Administration. 1979. OSHA Instruction CPL 2-2.20 (Subject: Industrial Hygiene Field Manual). Issued April 2, 1979. U.S. Department of Labor, Washington, DC.
- 11.2. American Public Health Association. 1977. Tentative Method of Analysis for Primary and Secondary Amines in the Atmosphere (Ninhydrin Method). Pp. 339-342 in M. Katz (ed.) Methods of Air Sampling and Analysis. American Public Health Association, Washington, DC.
- 11.3. USEPA. 1996. Method 8270C – Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS). SW-846 - Test Methods for Evaluating Solid Waste: Physical/Chemical Methods. Office of Solid Waste. Washington, DC. Online at http://www.epa.gov/epaoswer/hazwaste/test/8_series.htm. Accessed 3/3/2006.
- 11.4. USEPA. 1996. Method 3500B – Organic Extraction and Sample Preparation. SW-846 - Test Methods for Evaluating Solid Waste: Physical/Chemical Methods. Office of Solid Waste. Washington, DC. Online at http://www.epa.gov/epaoswer/hazwaste/test/3_series.htm. Accessed 3/3/2006.
- 11.5. USEPA. 1996. Method 8000B – Determinative Chromatographic Separations. SW-846 - Test Methods for Evaluating Solid Waste: Physical/Chemical Methods. Office of Solid Waste. Washington, DC. Online at http://www.epa.gov/epaoswer/hazwaste/test/8_series.htm. Accessed 3/3/2006.
- 11.6. USEPA. 1997. Conditional Test Method CTM-027 – Procedure for Collection and Analysis of Ammonia in Stationary Sources. Online at www.epa.gov/ttn/emc/ctm/ctm-027.pdf. Accessed 3/3/2006.
- 11.7. SOP A9. 2006. Measurement of Airflow Rate or Calibration of Air Sampling Instruments with Gilibrator 2 Calibration Systems. Standard Operating Procedure A9. Purdue Ag Air Quality Lab.
- 11.8. SOP B5. 2006. Management of Barn Data. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 11.9. SOP G4. 2006. Measurement of Ammonia (NH₃) Using the TEI Model 17C Chemiluminescence Analyzer. Standard Operating Procedure G4. Purdue Ag Air Quality Lab.
- 11.10. SOP G10. 2006. Measurement of Ammonia Using the MSA Photoacoustic Infrared (IR) Analyzer. Standard Operating Procedure G10. Purdue Ag Air Quality Lab.
- 11.11. SOP V5. 2006. Analysis and Quantitation of Amines by Ion Chromatography. Standard Operating Procedure V5. Purdue Ag Air Quality Lab.

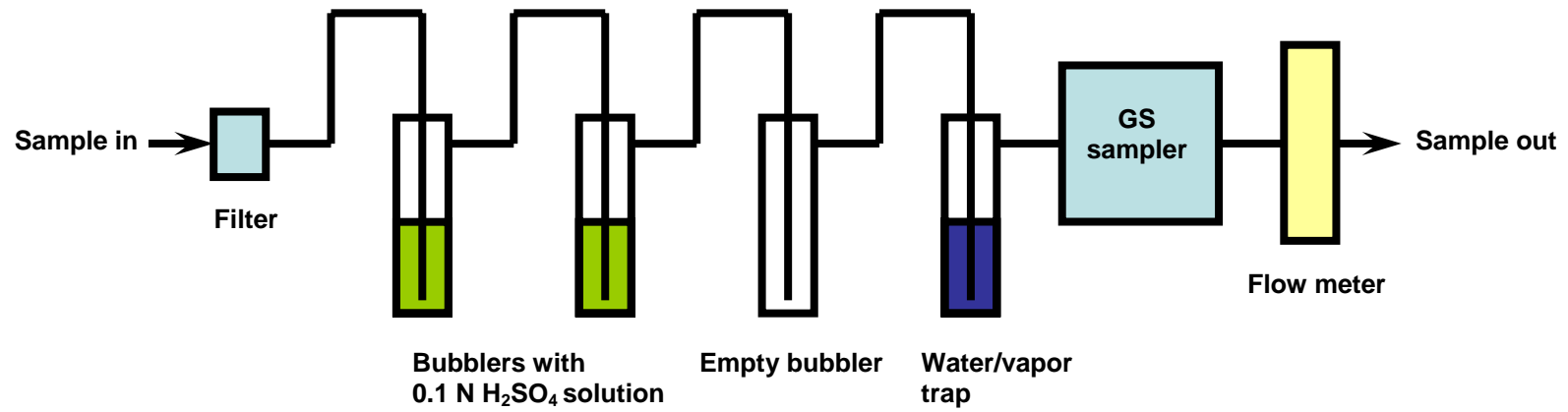


Figure 1. Proper configuration of the bubbler-based sampling train.

**THERMODESORPTION AND GC/MS ANALYSIS OF VOLATILE
ORGANIC**

COMPOUNDS (VOCs) COLLECTED ON SORBENT TUBES

Standard Operating Procedure (SOP) V4

**THERMODESORPTION AND GC/MS ANALYSIS OF VOLATILE ORGANIC
COMPOUNDS (VOCs) COLLECTED ON SORBENT TUBES
Standard Operating Procedure (SOP) V4**

**Prepared by
Bill W. Bogan and Hua Xu**

**Reviewed by
Albert J. Heber**

Effective Date: November 13, 2007

**PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)
Agricultural and Biological Engineering, Purdue University
West Lafayette, IN 47907**

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions	3
4. Health and Safety	5
5. Cautions	5
6. Interferences	6
7. Personnel Qualifications	7
8. Equipment and Supplies	7
9. Procedures	10
10. Data and Records Management	15
11. Quality Control and Quality Assurance	15
12. References	20

1. Scope and Applicability

- 1.1. This SOP describes a gas chromatography/mass spectrometry (GC/MS)-based method for detecting and quantitating volatile organic compounds (VOCs) in ambient air at livestock facilities.
 - 1.1.1. Specific VOC target compounds that are covered under this SOP are listed in Table 1 of SOP V1.
- 1.2. The VOCs in question will have been collected using sorbent tubes, as described in SOP V1; a necessary prerequisite for this analysis is therefore the desorption of the VOCs from the sampling tubes, which is accomplished using a thermal desorption unit.
- 1.3. The stated detection limit for this method is in the range of 0.5 to 25 parts per billion (ppbV) concentration in the air stream in question.
- 1.4. The methodology described in this SOP is derived from USEPA and ASTM methods (References 12.2, 12.6, 12.7, and 12.9)

2. Summary of Method

Sorbent tubes containing VOC analytes are collected according to SOP V1; the sampling matrix which is used (Carbotrap C/X) is particularly well-suited to the collection and retention of airborne VOCs. Tubes containing the samples are thermally desorbed, in what is referred to as the primary desorption step. Analytes driven off the tube in the primary desorption step are refocused on a secondary trap, which concentrates them into a very small volume. Rapid desorption of the trap (the secondary desorption step) follows, and then injection/transfer of target analytes into the GC. The above steps are accomplished using a GERSTEL TDS (ThermoDesorption System) 2 unit, and/or a GERSTEL TDS A 20-tube autosampler unit. The GC (Agilent Model 6890) accomplishes the separation of individual target analytes through the use of a Restek Rtx[®]-1 GC column. Identification of target compounds is accomplished by comparing their GC retention times and mass spectra (determined using an Agilent Model 5975 MSD) with those of authentic standards. Quantitation of each compound is then accomplished by comparing the response (preferably peak area of its primary characteristic ion, but peak height may also be used) relative to that of an internal standard. Suitable internal standards for this SOP include 1,4-dichlorobenzene-d₄, naphthalene-d₈, and acenaphthene-d₁₀.

3.

3. Definitions

- 3.1. **Thermal Desorption:** The use of heat and a flow of inert (carrier) gas to extract volatiles from a solid matrix directly into the carrier gas and transfer them to a downstream system element (in this case, the analytical column of a GC). No solvent is required.
- 3.2. **Two-stage Thermal Desorption:** The process of thermally desorbing analytes from a solid matrix, reconcentrating them on a focusing tube and then rapidly heating the focusing tube to “inject” the concentrated compounds into the GC system. This concentrates the analytes into a narrow band of vapor (equivalent to a small injection volume), which is compatible with high resolution capillary gas chromatography.

- 3.3. **Focusing Tube (Focusing Trap):** Narrow (typically <3-mm I.D.) tube containing a small bed of sorbent, which is maintained near or below ambient temperature and used to refocus and concentrate analytes which have been thermally desorbed from the sorbent tube. Once all the VOCs have been transferred from the sorbent tube to the focusing tube, the focusing tube is heated very rapidly to transfer the analytes into the capillary GC analytical column in a narrow band of vapor.
- 3.4. **Cryofocusing:** Use of coolant (for example, liquid nitrogen, liquid argon, or liquid CO₂) to lower the temperature of the focusing trap, promoting more rapid and complete sorption of analytes on the trap.
- 3.5. **Relative Retention Time (RRT):** The ratio of the retention time of an individual analyte over the retention time of the internal standard with which the analyte is being compared for purposes of identification. RRT is a unitless quantity, as the numerator and denominator time units cancel out. For identification of the analyte peak to be valid, RRT must be within 0.94 and 1.06 (i.e. agreement within 0.06 units).
- 3.6. **Response Factor (RF):** Instrument sensitivity towards a particular compound. The relationship between instrument output (e.g. peak height or integrated area) and analyte concentration.
- 3.7. **Relative Response Factor (RRF):** The comparison of the Response Factor of an analyte (i.e. a calibration standard, target compound, or surrogate) to that of an internal standard.
- 3.8. **MS-SCAN:** Mode of operation of a GC/MS in which all mass ions over a given mass range are swept over a given period of time.
- 3.9. **MS-SIM:** Mode of operation of a GC/MS in which only a single mass ion (or a selected number of discrete mass ions) are monitored.
- 3.10. **Total Ion Chromatogram (TIC):** Chromatogram produced from a mass spectrometer detector operating in full scan (MS-SCAN) mode.
- 3.11. **Extracted Ion Current Profile (EICP):** A plot showing the abundance of an ion of a particular mass, plotted as a function of retention time or scan number.
- 3.12. **Primary Characteristic Ion:** The particular ion, as defined by USEPA or other methods, which provides the best results in terms of quantitating a particular compound.
- 3.13. **Target Compound:** A compound designated by the QAPP or Statement of Work for analysis (qualitative or quantitative).
- 3.14. **Internal Standard (IS)** – A pure analyte which is added to a sample (or standard) solution in a known amount, and is used to measure the relative responses of target compounds and/or surrogates in the solution. The internal standard(s) must not already be present in the sample.
- 3.15. **Tuning Standard** – A standard which is used to (a) verify that the MS system is operating properly and (b) guard against reactive sites in the system (especially the injection ports, lines, and the first segments of the column) which might cause chemical breakdown or degradation of the sample.
- 3.16. **Surrogate Standard** – A compound that is chemically similar to the analyte group but is not expected to occur in an environmental sample. Surrogate(s) should be added to each sample, blank or standard just prior to processing. The recovery of the surrogate standard is then used to monitor for unusual matrix effects, gross sample processing

errors, etc. Surrogate recovery is evaluated for acceptance by determining whether the measured concentration falls within the acceptance limits.

- 3.17. **Laboratory Control Standard (LCS)** – A blank sample tube (identical to the tubes to be analyzed), to which known quantities of all method analytes (target compounds, internal standards and surrogates) are added in the laboratory. The LCS is analyzed exactly like a sample, and is used to assure that the system and analyst are capable of making accurate and precise measurements, and that there are no matrix effects (i.e. that analytes sorbed to the resin in sampling tubes are quantitated the same as those in solution).
- 3.18. **Method Blank** – A blank sample tube that is treated exactly as a sample, and is processed simultaneously with other samples in the analysis batch through all steps of the analytical procedure. This is done to determine if method analytes or other potential interferences are present in the lab environment, reagents, or apparatus.
- 3.19. **Calibration Check Compounds (CCC)** – A group of compounds used as a standard to evaluate the calibration from the standpoint of system integrity. Highly variable results with the CCCs may indicate system leaks or reactive sites on the column.
- 3.20. **System Performance Check Compounds (SPCC)** – SPCCs are compounds which are known to have very low RFs (0.1-0.2), and which tend to decrease rapidly in response as the chromatographic system (or the standard material) begins to deteriorate. They are usually the first to show poor performance, and are therefore used to ensure that the system is performing adequately.

4. Health and Safety

- 4.1. The red-anodized system parts in the TDS G and TDS A may become very hot, and may cause burns if touched. Particular care should also be exercised when opening the transfer chamber, as the inside of this chamber may reach 400°C. Always wait for the system to cool down before working on the transfer line.
- 4.2. Never operate the thermodesorption system in the presence of explosive fumes or gases.
- 4.3. Treat each chemical compound as a potential health hazard, and reduce exposure to chemicals as much as possible.
 - 4.3.1. Always wear protective clothing (gloves, lab coats, protective eyewear) when working with chemicals.
 - 4.3.2. Conduct all operations with solvents in properly-ventilated chemical fume hoods.
 - 4.3.3. Keep analytical balances and other work areas clean; deal with spills promptly.
- 4.4. Maintain a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method.
- 4.5. Also maintain a reference file of Material Safety Data Sheets (MSDSs), which will be made available to all personnel involved in these analyses.

5. Cautions

- 5.1. Contact between the capillary column and the wall of the GC oven can affect both chromatographic performance and column life. Do not allow the column to touch the oven walls.

- 5.2. Carrier gas should be scrubbed to remove traces of oxygen and scrubbers should be changed regularly. If the Agilent “big universal trap” (See Section 8.7.1) is used to purify helium, it should be changed after every 13th cylinder of ultra-high-purity helium is consumed.
- 5.3. The use of a guard column between the injection port and the analytical column may help prolong analytical column performance. Use guard columns (either as part of a pre-fabricated column (Section 8.3), or separate) when conducting analyses under this SOP.
- 5.4. When the online TDS G or TDS A is not in use, an empty tube must be inserted to avoid contamination of the CIS by airborne contaminants in the laboratory.
- 5.5. Do not operate the TDS G or TDS A in damp areas.

6. Interferences

- 6.1. Sample tubes awaiting analysis on an automated desorption system must be completely sealed before thermal desorption in order to prevent entry of VOC contaminants from the laboratory air, and/or losses of weakly retained analytes from the tube.
 - 6.1.1. Keep all tubes that are awaiting analysis individually wrapped in uncoated aluminum foil in an airtight container in a refrigerator $\leq 4^{\circ}\text{C}$.
 - 6.1.2. In no cases should the period between sampling and analysis exceed 30 d.
- 6.2. Use of other than inert materials (deactivated fused silica, glass lined tubing, glass, quartz and PTFE) in the sample flow path can result in degradation of some analytes.
- 6.3. Very volatile compounds can display peak broadening and co-elution with other species if the compounds are not delivered to the GC column in a small volume of carrier gas.
 - 6.3.1. Refocusing of the sample after collection on the primary trap (Section 9.3.7) mitigates this problem.
- 6.4. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of a humid zero air blank to check for cross-contamination.
- 6.5. When sample components are not resolved chromatographically, the result will be a mass spectrum containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s), or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.
- 6.6. When analytes coelute (i.e., only one chromatographic peak is apparent), the criteria for identification may be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound. In these cases, examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra and in qualitative identification of compounds.
- 6.7. Sorbent Artifacts
 - 6.7.1. Artifacts are typically < 1 ng per tube for well conditioned graphitized carbon sorbents.
 - 6.7.2. Stringent tube conditioning (See Section 8.1 of SOP V1) and careful tube handling procedures after conditioning are essential for minimizing these artifacts.

- 6.8. Water collected in the sorbent tubes during sampling can interfere with the subsequent analyses. Follow the approaches for minimizing the amount of water collected in sampling tubes that are given in SOP V1.
- 6.9. Compounds which are too labile or reactive for conventional GC analysis are not suitable for this method.
- 6.10. Several phenolic compounds outside the scope of this SOP (e.g. 4-nitrophenol, 2,4-dinitrophenol, 4,6-dinitro-2-methylphenol, and 4-chloro-3-methylphenol) can be subject to erratic chromatographic behavior when analyzed by this method; this is especially true if the GC system is contaminated with high-boiling-point material (high-molecular-weight, nonvolatile compounds).
 - 6.10.1. It is therefore possible that phenols relevant to this SOP may also display this behavior; care should thus be taken to keep the system free of contamination of this nature. The phenol-specific System Performance Check Compounds (SPCCs, Section 9.2.3.1) are run as standards to guard against this.

7. Personnel Qualifications

- 7.1. Personnel should be trained in the use of all applicable instrumentation, and in appropriate sample-handling procedures, before performing the activities in this SOP.
- 7.2. Each analyst must read applicable equipment manuals and this SOP before performing these activities.
- 7.3. Each individual analyst must demonstrate the ability to generate acceptable results with this method (Section 11.13).

8. Equipment and Supplies

- 8.1. Thermodesorption unit (GERSTEL Model TDS G Online Thermodesorption System) or thermodesorption autosampler (GERSTEL Model TDS A) with CIS 4 Cooled Injection System (CIS) as the interface between the TDS and gas chromatograph (GC).
- 8.2. Gas chromatograph: Agilent GC 6890 System (Agilent Technologies, Palo Alto, CA)
 - 8.2.1. This GC meets the required capabilities detailed in Methods 8270C and TO-15.
- 8.3. Column: Columns that are suitable for use under this SOP are the following:
 - 8.3.1. Rtx[®]-1 column capillary column 60m × 0.32 mm × 1.0 μm (Restek Corp., Part #10157).
 - 8.3.2. An equivalent column (or column/guard column combination) may be used if equal performance can be achieved.
- 8.4. Mass spectrometer: Agilent MSD 5975 System (Agilent Technologies, Palo Alto, CA)
 - 8.4.1. This MSD meets the required capabilities detailed in Methods 8270C and TO-15.
- 8.5. GC/MS Interface: Capillary-direct into the MS source
 - 8.5.1. This interface meets the required capabilities detailed in Methods 8270C and TO-15.
- 8.6. Data Analysis System: A computer system that allows continuous acquisition and storage of all mass spectra. Vendor (Agilent)'s software allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number (Extracted Ion Current Profile (EICP)). Vendor's software also integrates the abundances in any EICP between specified time or scan-number limits.

- 8.6.1. The most recent version of the EPA/NIST Mass Spectral Library will be utilized.
- 8.7. GERSTEL Adsorbent Tube Injector System (ATIS™)
- 8.8. Helium cylinders - 99.999% or higher purity He as the GC carrier gas
 - 8.8.1. Scrubber: The Agilent “big universal trap” (Part # RMSH-2), or equivalent should be used to purify the helium carrier gas.
- 8.9. Liquid N₂ for cooling the thermodesorption unit’s Cooled Inlet System
- 8.10. Standards
 - 8.10.1. Stock standard solutions
 - 8.10.1.1. The identity of chemicals to be used as standards will be dependent on the specific target analyte class in a particular analytical run. This information will be included in the QAPP and/or Statement of Work. General procedural steps common to all standards are given in the following sections.
 - 8.10.1.2. Prepare stock standard solutions by weighing (on an analytical balance capable of weighing 0.0001 g) about 0.0100 g of pure material. Dissolve the compound in pesticide-quality solvent and dilute to volume in a 10-mL volumetric flask.
 - 8.10.1.2.1. Use methanol whenever possible, as it is considerably more volatile than most target analytes, and can be purged from the tube (See Step 8.10.2.4) after spiking with minimal (or no) loss of analyte.
 - 8.10.1.2.2. In cases where methanol use and purging is not feasible (i.e. very volatile target analytes, compounds which are insoluble in methanol) use ethyl acetate instead, as it is very readily resolved chromatographically from most target analytes.
 - 8.10.1.2.3. When compound purity is assayed to be 96% or greater, use weights without correction to calculate the concentration of the stock standard. Otherwise, use appropriate correction factors to calculate analyte stock concentrations.
 - 8.10.1.2.4. Commercially-prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.
 - 8.10.1.3. Store stock standard solutions in bottles with PTFE-lined screw-caps. They should be protected from light, and maintained at -10°C (or less if so recommended by the standard manufacturer).
 - 8.10.1.4. Check stock standard solutions frequently for signs of degradation or evaporation, especially just prior to using them to prepare calibration standards.
 - 8.10.1.5. Replace stock standard solutions after one year.
 - 8.10.2. Calibration standards
 - 8.10.2.1. Prepare calibration standards at a minimum of five different concentrations (e.g. a minimum of a five-point calibration curve). Each standard should contain each target compound which is intended to be detected/quantified by this method.
 - 8.10.2.1.1. The calibration standards should correspond to the range of concentrations found in actual samples but should not exceed the range of the GC/MS system.
 - 8.10.2.2. Load the calibration standards onto clean tubes using the GERSTEL ATIS system. Turn on nitrogen cylinder connected to the ATIS.
 - 8.10.2.3. Turn the flow controller valve to “ON”.
 - 8.10.2.4. Flip the power switch to “High”(50 °C ~ 120 °C).

- 8.10.2.5. Check the thermometer to verify that the temperature reaches $>10^{\circ}\text{C}$ above the boiling point of the solvent used to prepare the standards. It may take a couple of hours to reach the desired temperature.
 - 8.10.2.6. Loosen the white wheel on the left side of the unit, insert a sorbent tube (paying attention to the flow direction indicated on the tube), and tighten the white wheel.
 - 8.10.2.7. Connect the Alltech digital flowmeter at the end of the tube to check the flow rate ($\sim 50 \text{ mL/min}$). Use flexible Tygon tubing, with a short piece of 6-mm ID PFE tubing as the connection between the Tygon tubing and sorbent tube.
 - 8.10.2.8. Adjust the flow controller valve to $\sim 50 \text{ mL/min}$
 - 8.10.2.9. Use the microsyringe to inject standards into the ATIS.
 - 8.10.2.9.1. Change the green septum every 25 injections to maintain a good seal
 - 8.10.2.10. Do not inject more than 25 μL of the standard solution.
 - 8.10.2.11. If the liquid standard has been prepared in a solvent which will pass through the sorbent while analytes are quantitatively retained, do not disconnect the tube from the injector until the solvent has been eliminated from the sorbent bed (approximately 5 min under the conditions specified).
 - 8.10.2.11.1. In cases where the solvent can be purged from the tube while quantitatively retaining the analytes, make a 5-10 μL injection (Step 8.10.2.2), as greater accuracy is possible with larger volumes.
 - 8.10.2.11.2. If the solvent cannot be purged from the tube, keep the injection volume in Step 8.10.2.2 as small as possible (0.5 - 1.0 μL), to minimize solvent interference in the GC analysis.
 - 8.10.2.12. Loosen the white wheel and take the sorbent tube out. Immediately cap it and place it in an appropriate storage container.
 - 8.10.2.13. Do not store tubes containing calibration standards more than 30 d prior to use.
- 8.10.3. Internal standards
- 8.10.3.1. Choose internal standards such that most of the analytes of interest in a chromatogram will have retention times of 0.80-1.20 relative to one (or more) of the internal standards.
 - 8.10.3.2. Because 1,4-dichlorobenzene- d_4 , naphthalene- d_8 , and acenaphthene- d_{10} are each appropriate as internal standards with some phenols (but not others) according to Method 8270C, all three should be used as internal standards when running samples that are expected to contain multiple phenolic compounds and VFAs.
 - 8.10.3.2.1. Other compounds that are used as surrogates in Method 8270C, and therefore may be of some use as internal standards under the present method, include phenol- d_6 , 2-fluorophenol, 2,4,6-tribromophenol, nitrobenzene- d_5 , 2-fluorobiphenyl, and/or *p*-terphenyl- d_{14} .
 - 8.10.3.3. Dissolve 0.200 g of each compound with a small volume of methanol. Transfer to a 50-mL volumetric flask and dilute to volume with methanol.
 - 8.10.3.3.1. As described in Section 8.10.1.2.2, ethyl acetate may be substituted for methanol if the particular compound(s) are insufficiently soluble in methanol.
 - 8.10.3.3.2. The resulting solution contains each standard at a concentration of 4,000 $\text{ng}/\mu\text{L}$.

- 8.10.3.4. Following the procedures in Section 8.10.2, spike each sample tube which is to be analyzed with 10 μL of the internal standard solution, such that approximately 100 ng of each internal standard compound is introduced to the tube.
- 8.10.3.5. Area counts of the internal standard peaks should be between 50-200% of the areas present in the midpoints of the calibration curves for each target compound.
- 8.10.3.6. Store internal standard mixtures at -10°C (or below) when not in use.
- 8.10.3.7. Prepare fresh internal standard mixtures once a year.
- 8.10.3.8. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

9. Procedures

- 9.1. Set up the GC/MS, and establish the operating conditions. Method development at PAAQL has yielded the following recommendations for setting the various parameters:

Mass range	34-265 amu
Scan time	5.84 s/scan
Initial temperature	35°C , hold for 1 min
Temperature program	$35-100^{\circ}\text{C}$ at $10^{\circ}\text{C}/\text{min}$, $100-250^{\circ}\text{C}$ at $25^{\circ}\text{C}/\text{min}$
Final temperature	250°C , hold for 10 min
Injector	Cooled Injection System (CIS)*
Transfer line temperature	270°C
Thermodeportion final temperature	250°C
CIS final temperature	280°C
MS interface temperature	280°C
Carrier gas	Helium (22 cm/s)
Ion trap only	Set axial modulation, manifold temperature, and emission current to manufacturer's recommendations

*Cooled injection system is used as the inlet for the GC associated with TDS.

- 9.2. Initial tuning, initial calibration, and system performance check
- 9.2.1. Initial tuning and calibration
- 9.2.1.1. Use Perfluorotributylamine (PFTBA) as the tuning compound along with the 5975's built-in calibrant delivery system to perform Autotune (standard spectra tune) as initial tuning or after any maintenance.
- 9.2.1.2. Perform quick tune daily, at the beginning of each batch of samples (i.e. once per 24-h time period of operation).
- 9.2.1.3. Use the following as tuning acceptance criteria for the PFTBA standard:
- 9.2.1.3.1. The ratio of the ion at m/z 69 to that at m/z 70 must be $1 \pm 0.50\%$.
- 9.2.1.3.2. The ratio of the ion at m/z 219 to that at m/z 220 must be $5 \pm 1.25\%$.
- 9.2.1.3.3. The ratio of the ion at m/z 502 to that at m/z 503 must be $10 \pm 1.0\%$.
- 9.2.1.3.4. The peak width at half-height for the ions at m/z 69, 219, and 502 must be 0.5 ± 0.2 amu.

- 9.2.1.3.5. The mass assignments for the ions at m/z 69, 219, and 502 must be correct to within 0.1 amu (e.g., 69.0 ± 0.1 amu).
 - 9.2.1.4. Alternatively, use other documented tuning criteria (e.g. manufacturer's instructions), provided that performance is not adversely affected.
 - 9.2.1.5. Special Autotune can be performed to meet the tuning criteria of decafluorotriphenylphosphine (DFTPP) and 4-bromofluorobenzene (BFB) required by EPA for semivolatiles and volatiles.
 - 9.2.1.6. Do not begin analyses until the tuning criteria are met.
 - 9.2.2. System performance check
 - 9.2.2.1. Perform a system performance check daily to ensure that minimum average RFs are met before the calibration curve is used. For this SOP, the System Performance Check Compounds (SPCCs) are: chloromethane, 1, 1-dichloroethane, bromoform, chlorobezene, and 1,1,2,2-tetrachloroethane (volatitiles);N-nitroso-di-n-propylamine; hexachlorocyclopentadiene; 2,4-dinitrophenol; and 4-nitrophenol (semivolatiles).
 - 9.2.2.2. These SPCCs typically have very low RFs (0.1-0.2) and tend to decrease in response as the chromatographic system (or the standard material) begins to deteriorate. They are usually the first to show poor performance. Calculate RF for each of the four SPCCs; the minimum acceptable average RF is 0.050. This check must be met before sample analysis begins.
 - 9.2.2.3. If the minimum response factors are not met, evaluate the system and take corrective action before sample analysis begins. Possible problems include:
 - 9.2.2.3.1. Standard mixture degradation
 - 9.2.2.3.2. Injection port inlet contamination
 - 9.2.2.3.3. Contamination at the front end of the analytical column
 - 9.2.2.3.4. Reactive (i.e. non-inert) sites in the column and/or system
 - 9.2.3. Perform the daily calibration check (Section 11.6) after the GC/MS system has met the tuning criteria for PFTBA, but before any actual samples are run.
- 9.3. Sample analysis
 - 9.3.1. Remove the sampling tubes (including any backup tubes that were used in the sampling event), as well as all necessary blanks, from their storage container and allow the tubes to come to room temperature. Always wear clean gloves when handling tubes.
 - 9.3.2. Spike each tube with internal standard. See Section 8.9.3.
 - 9.3.3. Load the tubes into the thermal desorption unit.
 - 9.3.3.1. If using the TDS G desorption unit, the procedures are as follows:
 - 9.3.3.1.1. Free the receiving member by pushing the locking member to the left.
 - 9.3.3.1.2. Pull the receiving member out of the TDS oven.
 - 9.3.3.1.3. Pull the TDS tube last used out of the receiving member.
 - 9.3.3.1.4. Push a new TDS tube containing the sample onto the receiving member.
 - 9.3.3.1.5. Push the receiving member and the inserted TDS tube into the TDS oven as far as it will go.
 - 9.3.3.1.6. Lock the online TDS G by pushing the locking member to the right again.
 - 9.3.3.2. If using the TDS A autosampler, the procedures are as follows.
 - 9.3.3.2.1. Insert the TDS tube into the sample magazine, as in Fig. 1.

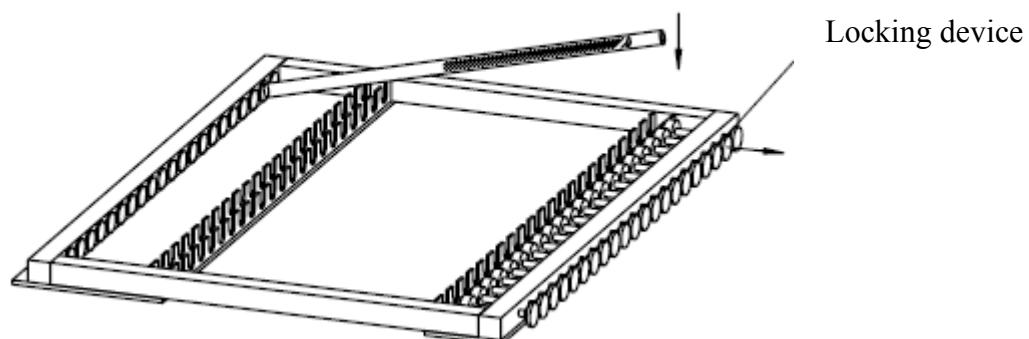


Figure 1. Inserting a sample tube into the tray of the TDS A autosampler.

- 9.3.3.2.2. Insert the left-hand side of the tube with the left hand into the left-side of the sample magazine.
- 9.3.3.2.3. Pull the appropriate locking device against spring pressure to the right with the right hand.
- 9.3.3.2.4. Introduce the tube completely with the left hand and release the locking device.
- 9.3.4. Check for TDS leaks
 - 9.3.4.1. Perform leak checks after the system has been serviced, and after each new installation of the system.
 - 9.3.4.2. The following symptoms indicate leaks in the system:
 - 9.3.4.2.1. After the TDS has been opened and closed, it takes longer than usual to build up the head pressure.
 - 9.3.4.2.2. The head pressure is not maintained during splitless transfer or at the end of the oven program.
 - 9.3.4.2.3. Sensitivity and reproducibility are reduced.
 - 9.3.4.3. First, conduct a rapid check of system flows.
 - 9.3.4.3.1. Set the GC to Splitless mode.
 - 9.3.4.3.2. Total flow is the sum of column flow, septum purge flow and TDS purge flow.
 - 9.3.4.3.3. If total flow is significantly higher than the expected sum of the individual flows, there is either a leak or the flows are not set properly.
 - 9.3.4.3.4. If these flows are properly set and actual flow is higher than the set flow, there is probably a leak.
 - 9.3.4.4. An electronic leak detector may subsequently be used to check all connections for leaks.
 - 9.3.4.5. Halt the run if any leaks are detected in the TDS.
- 9.3.5. Primary desorption
 - 9.3.5.1. Set the flow rate of UHP He gas to at least 20 mL/min, in the reverse direction to that of the air flow during sampling. Analytes are thus desorbed from the tube in “backflush” mode.

- 9.3.5.2. Heat the tube up to 325°C (use 250 °C)with a heating rate from 1°C/min to 60°C/min.
- 9.3.5.3. Set a transfer line temperature at least 20°C above the highest desorption temperature. Typically, the transfer line is held between 250°C ~ 350°C. (use 270 °C)
- 9.3.5.4. VOCs are transferred onto the GERSTEL Cooled Injection System (CIS), which functions as both a focusing trap and a GC inlet. Thermal desorption continues until all target species are transferred to the focusing trap (the liner of the inlet).
- 9.3.6. Secondary desorption
- 9.3.6.1. After the primary desorption step is complete, rapidly (e.g. a ramp of 40°C) heat the focusing trap to 325°C (use 280 °C), and apply a flow of at least 3 mL/min of dry UHP helium carrier gas.
- 9.3.6.2. Desorb analytes from the focusing trap in “backflush” mode, with the gas flow through the trap in the opposite direction from that used during the focusing step.
- 9.3.6.3. Choose the proper CIS inlet mode (solvent vent is strongly recommended) and set the right split ratio and split flow in the GC Chemstation.
- 9.3.6.4. The GC oven program starts when CIS program begins.
- 9.3.7. GC/MS analysis
- 9.3.7.1. Appropriate operating conditions for capillary GC columns used under this SOP are as follows:

Initial temperature	35°C, hold for 1 min
Temperature program	35-100°C at 10°C/min 100-250°C at 25°C/min
Final temperature:	220°C, hold for 3 min
Carrier gas	Helium (22 cm/s)

- 9.3.7.2. Operate the GC/MS system to scan the atomic mass range from 34 to 265 amu.
- 9.3.7.3. Acquire at least ten scans per chromatographic peak.
- 9.3.7.4. Selected ion monitoring (SIM) is acceptable for applications requiring detection limits below the normal range of electron impact mass spectrometry. However, SIM may provide a less definitive compound identification, unless multiple ions are monitored for each analyte.
- 9.3.7.5. Acceptance criteria for samples are as follows:
- 9.3.7.5.1. If the retention time for any internal standard component changes by more than 20 s relative to the latest daily calibration check standard, inspect the GC/MS system for malfunctions and make corrections as required.
- 9.3.7.5.2. If the area response (Y) for the primary ion in any internal standard component changes by more than ±40% relative to the latest daily calibration check standard, inspect the GC/MS system for malfunctions and make corrections as required.
- 9.3.7.5.3. Mark any samples that fail these criteria as invalid data, as re-running of the same samples under this SOP is not possible.

9.3.8. Qualitative analysis (peak identification)

- 9.3.8.1. In keeping with EPA Method 8270C, base qualitative identification of compounds upon (a) retention time and (b) comparison of the sample mass spectrum (after any necessary background correction) with a reference mass spectrum. Conditions which must be met to identify compounds are as follows:
- 9.3.8.1.1. The relative retention time (RRT) of the sample component must be within ± 0.06 RRT units of the RRT of the standard component.
- 9.3.8.1.2. The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. For the purposes of this SOP, peak selection is done by a data system target compound search routine based on the presence of a target peak, containing ions specific for the target compound, at a compound-specific retention time.
- 9.3.8.1.3. The characteristic ions from the reference mass spectrum are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum.
- 9.3.8.2. The relative intensities of the characteristic ions in the spectrum of the sample component must agree to within 30% of the relative intensities of the same ions in the reference spectrum.
- 9.3.8.2.1. For example: If an ion has an abundance of 50% in the reference spectrum, the abundance of the same ion in a sample spectrum must be 20% -80%.
- 9.3.8.3. Structural isomers that produce very similar mass spectra should be identified as individual isomers only if they have sufficiently different GC retention times.
- 9.3.8.3.1. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights.
- 9.3.8.3.2. If this criterion is not met, structural isomers should be identified as isomeric pairs.
- 9.3.8.4. For samples containing components not associated with the calibration standards (e.g. unknown or unidentified components), a library search may be made for the purpose of tentative identification. Guidelines for tentative identification are as follows:
- 9.3.8.4.1. The molecular ion(s) and all major ions in the reference spectrum (ions > 10% of the most abundant ion) should also be present in the sample spectrum.
- 9.3.8.4.2. The relative intensities of the major ions should agree within $\pm 20\%$. As an example, an ion with an abundance of 50% in the spectrum of the standard must have an abundance in the sample of between 30 and 70%.
- 9.3.8.4.3. Ions present in the sample spectrum but not in the reference spectrum should be reviewed to determine if they may be due to background contamination or the presence of coeluting compounds.
- 9.3.8.4.4. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

9.3.9. Quantitative analysis

- 9.3.9.1. Once a compound has been identified, base its quantitation upon the integrated abundance of the primary characteristic ion from the EICP.

- 9.3.9.2. Where applicable, estimate the concentration(s) of any non-target analyte(s) identified in the sample by using the formula given in Section 11.5.6.
 - 9.3.9.2.1. Take the areas A_x and A_{is} from the total ion chromatograms, and assume the RF for the compound to be 1.
 - 9.3.9.2.2. Use the nearest internal standard which is free of interference.
 - 9.3.9.2.3. Report all concentrations derived in this way as estimates, and note which internal standard was used in the calculation for each analyte.
 - 9.3.9.3. Structural isomers that produce very similar mass spectra should be quantitated as individual isomers if they have sufficiently different GC retention times.
 - 9.3.9.3.1. Sufficient GC resolution is defined as the height of the valley between two isomer peaks being less than 25% of the sum of the two peak heights.
 - 9.3.9.4. Quantitate structural isomers that do not meet this criterion for separation together as isomeric pairs.
 - 9.3.9.5. Quantitative results (mass of analyte per tube) must be divided by sample volume (See SOP V1) to determine concentration in the original air sample.
- 9.4. Restoring the sorbent tubes
 - 9.4.1. A proper thermodesorption step will leave the tubes ready for re-use.
 - 9.4.2. Remove the tubes from the thermal desorber.
 - 9.4.3. Using clean gloves, rewrap the tubes with aluminum foil, replace in the clean, airtight container, and re-store the tubes in a cool environment ($\leq 4^\circ\text{C}$) until their next use.
 - 9.4.4. See SOP V1 for information as to how long tubes may be stored without needing partial or full re-conditioning. Conditioning of tubes is done with the GERSTEL Model TC1 Tube Conditioner.

10. Data and Records Management

- 10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
- 10.2. Manage all data according to SOP B5.
- 10.3. Document all data and information on laboratory data sheets, and within logbooks with permanent ink, or in electronic notes files.
- 10.4. Overstrike all errors in writing with a single line. Initial and date all such corrections.

11. Quality Control and Quality Assurance

- 11.1. Carrier gas
 - 11.1.1. Inert, 99.999% or higher purity helium will be used as carrier gas.
 - 11.1.2. All carrier gas lines will be either stainless steel or copper.
 - 11.1.3. Install "big universal trap" scrubber (See Section 8.8.1) on the carrier gas lines supplying the analytical system. Replace these filters regularly, according to the manufacturer's instructions (i.e. after every 13th He cylinder).
- 11.2. Reagent purity
 - 11.2.1. Use only reagent-grade inorganic chemicals.

- 11.2.2. Unless otherwise indicated, ensure that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.
- 11.2.3. If any other grade of reagent is to be used, it must be first ascertained that it is sufficiently pure to maintain the accuracy of the determination.
- 11.2.4. Use only pesticide-quality (or equivalent) solvents.
- 11.3. Tube labeling and identification must follow Section 10.1 of SOP V1.
- 11.4. Compatibility between sorbent tubes and desorption unit
 - 11.4.1. The position and length of the sorbent bed must be such that no part of the sorbent bed extends outside that portion of the tube which is directly heated by the thermal desorption oven. Only use tubes that meet this criterion.
- 11.5. Five-point (full) calibration
 - 11.5.1. Do a full calibration during initial instrument setup, whenever the analytical method parameters are substantially changed, or whenever significant maintenance is performed on the system. Significant maintenance includes:
 - 11.5.1.1. Ion source cleaning or repair
 - 11.5.1.2. Injection port cleaning (e.g. change septa, O-ring, liner, and/or gold-plated seal)
 - 11.5.1.3. Column replacement
 - 11.5.2. Do a full calibration whenever the system fails the daily single-point calibration check (Section 11.6) twice in the same day.
 - 11.5.3. Calibrate the system at a minimum of five concentrations that span the monitoring range of interest. The lowest point on the calibration curve must be below the lowest concentration which meets the data quality objectives of the project, and should be close to the lower detection limit for the analyte in question. One point of the full calibration curve must correspond to the concentration in the daily single-point calibration check standard (Section 11.6).
 - 11.5.4. Add internal standard with each point of the calibration curve. The volume and mass of internal standard added must be the same for all calibration samples, and must be the same mass and volume added for all samples.
 - 11.5.5. Use the primary ions for each compound to quantitate all analytes in the calibration curve, unless interferences are present, in which case use a secondary ion.
 - 11.5.6. Calculate relative response factors (RRFs) for each target compound in each of the calibration standards, relative to the internal standard component with the closest retention time, according to the following equation:

$$RRF = \frac{A_s \times M_{is}}{A_{is} \times M_s}$$

Where:

A_s = Peak area (or height) of the analyte or surrogate

A_{is} = Peak area (or height) of the internal standard

M_s = Mass of the analyte or surrogate (ppbV)

M_{is} = Mass of the internal standard (ppbV)

- 11.5.6.1. Calculate mean RRF (using each of the five concentrations in the standard curve), as well as the standard deviation and %RSD.
- 11.5.7. Calculate relative retention times (RRTs) for each target compound in each of the calibration standards, relative to the internal standard component with the closest retention time, according to the following equation:

$$\text{RRT} = \frac{\text{RT}_c}{\text{RT}_{is}}$$

Where:

RT_c = Retention time of the target compound

RT_{is} = Retention time of the internal standard

- 11.5.8. Calculate mean RRT (using each of the five concentrations in the standard curve), as well as the standard deviation and %RSD.
- 11.5.9. Calculate mean retention time (RT), using each of the five concentrations in the standard curve.
- 11.5.10. Tabulate the area response (Y) of the primary ion for each compound and the corresponding mass for each compound and internal standard.
- 11.5.11. Calculate the mean area response for each internal standard compound over the entire calibration range.
- 11.5.12. Technical Acceptance criteria for the initial calibration are as follows:
- 11.5.12.1. The calculated %RSD for the RRF for each compound in the calibration table must be less than 30%. There may be at most two exceptions with %RSDs up to 40%. No compounds may have an RRF %RSD greater than 40%.
- 11.5.12.2. The RRT for each target compound at each individual calibration level must be within 6% of the mean RRT for the compound.
- 11.5.12.3. For each internal standard component, the area response Y of each target compound's primary ion at each calibration level must be within 40% of the mean area response for that ion over the entire calibration range.
- 11.5.12.4. For each internal standard component, the retention time at each calibration level must be within 20 s of the mean retention time over the initial calibration range.
- 11.5.13. Initial calibration acceptance criteria must be met before analysis of actual field samples is initiated.
- 11.5.13.1. If the initial calibration technical acceptance criteria are not met, inspect the system for problems. It may be necessary to clean the ion source, change the column, or take other corrective actions.
- 11.5.14. Do not report quantitative results for target compound(s) not included in the calibration standard(s), unless they are estimated (and denoted as such) according to Section 9.3.10.2.
- 11.5.15. Keep a control chart (SOP Q1) of calibration curve results, so that they may be compared over time to assess possible detector drift or other biases/inaccuracies that may develop over time. If the average peak area per unit concentration of any given analyte drifts from its baseline value established at the beginning of the study by more than ±10%, troubleshoot the system to identify and rectify the problem.

11.6. Daily (single-point) calibration checks

- 11.6.1. Perform a check of the calibration curve once every 24 h – after the GC/MS system has met the tuning criteria for PFTBA, but before any actual samples are run.
- 11.6.2. Analyze the mid-level calibration standard (with internal standards added) for the calibration check.
- 11.6.3. Perform the following calculations:
 - 11.6.3.1. Calculate a relative response factor (RRF) for each target compound using the equation in Section 10.5.6.
 - 11.6.3.2. Calculate the percent difference (%D) in the RRF of the daily RRF compared to the mean RRF in the most recent full calibration. Calculate the %D for each target compound using the following equation:

$$\%D = \frac{(\text{RRF}_c - \overline{\text{RRF}}_i)}{\overline{\text{RRF}}_i} \times 100$$

Where:

RRF_c = RRF of the target compound in the daily calibration check run

$\overline{\text{RRF}}_i$ = Mean RRF of the target compound in the most recent full calibration

- 11.6.4. Acceptance criteria for daily calibration checks are as follows:
 - 11.6.4.1. The %D for each target compound in a daily calibration sequence must be within $\pm 30\%$ in order to pass the calibration check and proceed with the analysis of samples and blanks.
 - 11.6.4.2. A control chart showing %D values for each daily calibration check will be maintained. Values for %D which are steadily increasing over time (but still within the acceptable range) may be an indication that maintenance will be needed in the future.
- 11.6.5. If the daily calibration technical acceptance criteria are not met, inspect the system for problems. It may be necessary to clean the ion source, change the column, or take other corrective action(s).
 - 11.6.5.1. Rerun the daily calibration check standard. If the %D criterion still cannot be met, more thorough corrective action may be needed.
 - 11.6.5.1.1. In this case, rerun the full calibration (Section 11.5).
- 11.7. Conduct a calibration check standard approximately every tenth sample during an extended analytical sequence, as a check on system performance. (EPA Method TO-15)
 - 11.7.1. For runs containing more than 15 samples, a calibration check standard may be inserted near the middle of the run, and evaluated as per Section 11.6.3. For runs longer than approximately 30 samples, conduct two calibration checks.
- 11.8. Check each analytical run for saturation. The level at which an individual compound will saturate the detection system is a function of the overall system sensitivity and the mass spectral characteristics of that compound. Denote any analyte(s) in a particular sample that are found to be saturated as “above the quantitation limit” (with the limit identified) in that sample.
- 11.9. Use secondary ion quantitation only when there are sample matrix interferences with the primary ion.

- 11.9.1. If secondary ion quantitation is performed, document the reasons for doing so.
- 11.10. Blanks
- 11.10.1. Lab blanks must be identically prepared tubes, from the same batch, with similar histories and conditioned at the same time as tubes used for sample collection.
- 11.10.1.1. Include at least two lab blanks per sampling event.
- 11.10.1.2. Store lab blanks in the lab in clean controlled conditions prior to use.
- 11.10.1.3. Analyze lab blanks at the same time as the samples (after the calibration check) - one at the beginning and one at the end of the sequence of runs.
- 11.10.1.4. Add internal standard to lab blanks in the same mass and volume added to samples.
- 11.10.1.5. Acceptance criteria for laboratory blanks are as follows:
- 11.10.1.5.1. The area response (Y) for the primary ion of each internal standard component in the blank must be within $\pm 40\%$ of the mean area response (Section 10.5.11) of the IS in the most recent valid full calibration.
- 11.10.1.5.2. The retention time for each of the internal standard components must be within ± 20 s of the MRT in the most recent valid full calibration.
- 11.10.1.5.3. The blank should not contain any target analyte at a concentration greater than its quantitation level (three times the MDL, as defined in Section 10.11).
- 11.10.1.5.4. The blank should also not contain any additional (non-target) compounds with retention times and mass-spectral features that would interfere with identification and measurement of a target analyte.
- 11.10.1.6. If the blanks do not meet the technical acceptance criteria, investigate the source of the contamination and take (and document) appropriate corrective measures before proceeding with further sample analysis.
- 11.10.1.7. Whenever a particularly concentrated sample is encountered (i.e., one outside the calibration range), run a blank tube immediately after the sample is completed, as a check against carryover effects.
- 11.10.2. Field blanks are the same as lab blanks. However, field blanks are transported to and from the monitoring site, and are uncapped and immediately resealed at the monitoring site. They do not actually have air pumped through them.
- 11.10.2.1. Take one field blank for every ten tubes included in a sampling event. No less than two field blanks may be included, however small the study.
- 11.10.2.2. Distribute field blanks evenly throughout the run when the tubes are analyzed.
- 11.11. Method detection limits
- 11.11.1. Determine the method detection limit for each compound on a particular GC/MS system by making seven replicate measurements of the compound of interest at a concentration near (within a factor of five) the expected detection limit. Calculate the standard deviation for the seven replicate concentrations, and multiply it by 3.14 (the Student's *t* value for 99% confidence for seven values).
- 11.12. System precision checks
- 11.12.1. Test the precision of the analytical system using six calibration check standard tubes.
- 11.12.2. Conduct system precision checks once every tenth batch of samples or once every three months, whichever happens first.

- 11.12.3. Conduct system precision checks whenever the analytical method is changed or modified.
- 11.12.4. Include the report produced from the most recent precision test with the report generated for each batch of samples.
- 11.13. Initial demonstration of analyst proficiency
 - 11.13.1. Whenever new analysts are trained to conduct the analyses described in this SOP, they will be required to successfully complete all of the calibrations, performance checks, and verifications outlined in this SOP prior to generating actual data.
- 11.14. Modifications or changes to the system
 - 11.14.1. If any changes are made to the system (e.g., the column is changed, a septum is changed), the following guidance (from Section 8.2 of USEPA Method 8000B) dictates whether or not recalibration of the system is necessary.
 - 11.14.1.1. The following procedures will automatically require recalibration of the system:
 - Changing, replacing, or reversing the column
 - Cleaning the MS source
 - Changing the electron multiplier, ion source chamber, or injector port
 - 11.14.1.2. The following procedures will probably not require recalibration of the system, but must still be followed by a calibration check:
 - Changing compressed gas cylinders and/or PTFE transfer lines
 - Changing septa or split seals
 - Changing syringes
 - Changing moisture, hydrocarbon, or oxygen traps
 - Changing column fittings, inlets, or filaments
 - Cleaning the inlet port
 - Breaking off or changing a guard column

12. References

- 12.1. Agilent MSD ChemStation Help. 2005. Overview of MSD Productivity ChemStation. Agilent Technologies, Inc. Palo Alto, CA.
- 12.2. ASTM D 6196-03. 2003. Standard Practice for Selection of Sorbents, Sampling, and Thermal Desorption Analysis Procedures for Volatile Organic Compounds in Air. ASTM International, West Conshohocken, PA US.
- 12.3. TDS 3 Manual. 2006. Operation Manual – Thermodesorption System TDS 3. GERSTEL, Inc., Mulheim an der Ruhr, Germany.
- 12.4. TDS A Manual. 1996. Operation Manual – Thermodesorption System TDS 2 with Autosampler TDS A in Conjunction with a GC HP6890 and a Gerstel Cooled Injection System CIS 4. GERSTEL, Inc., Mulheim an der Ruhr, Germany.
- 12.5. TDS G Manual. 2000. Operation Manual - TDS G Online Thermodesorption System in conjunction with a GC HP6890 and a GERSTEL CIS 4 Cold Injection System. GERSTEL, Inc., Mulheim an der Ruhr, Germany.
- 12.6. EPA Method TO15 1999. Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS). Compendium Method TO-15: In Compendium of Methods for

- the Determination of Toxic Organic Compounds in Ambient Air, Second Edition (EPA/625/R-96/010b). Center for Environmental Research Information, Office of Research and Development, Cincinnati, OH.
- 12.7. EPA Method TO17. 1999. Determination of Volatile Organic Compounds in Ambient Air Using Active Sampling Onto Sorbent Tubes. In Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition (EPA/625/R-96/010b). Compendium Method TO-17: Center for Environmental Research Information, Office of Research and Development, Cincinnati, OH.
 - 12.8. EPA Method 8081A. 1996. Organochlorine Pesticides by Gas Chromatography. SW-846 - Test Methods for Evaluating Solid Waste: Physical/Chemical Methods. Office of Solid Waste. Washington, DC. [http://www.epa.gov/epaoswer/hazwaste/test/8_series.htm.]
 - 12.9. EPA Method 8270C. 1996. Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS). SW-846 - Test Methods for Evaluating Solid Waste: Physical/Chemical Methods. Office of Solid Waste. Washington, DC. [http://www.epa.gov/epaoswer/hazwaste/test/8_series.htm.]
 - 12.10. CDPHE. 2000. Guidance for Analysis of Indoor Air Samples. Hazardous Materials and Waste Management Division Colorado Department of Public Health and the Environment, Denver, CO. [<http://www.cdphe.state.co.us/hm/airsmpl.pdf>]. Accessed 3/30/2006.
 - 12.11. Cai, L., J.A. Koziel, Y.-C. Lo, and S.J. Hoff. 2006. Characterization of volatile organic compounds and odorants associated with swine barn particulate matter using solid-phase microextraction and gas chromatography-mass spectrometry-olfactometry. *J. Chromatography A* 1102:60-72.
 - 12.12. McGinn, S.M., H.H. Janzen and T. Coates. 2003. Atmospheric ammonia, volatile fatty acids, and other odorants near beef feedlots. *J. Environmental Quality* 32:1173-1182.
 - 12.13. Wright, D.W., D.K. Eaton, L.T. Nielsen, F.W. Kuhrt, J.A. Koziel, J.P. Spinhirne, and D.B. Parker. Multidimensional gas chromatography-olfactometry for the identification and prioritization of malodors from confined animal feeding operations. *Journal of Agricultural and Food Chemistry* 53:8663-8672.
 - 12.14. Alltech Associates. 1997. Phenols by Heliflex® AT-1. Accessed 2/22/2006. [http://www.alltechweb.com/chromaccess/chromview_prn.asp?refno=1256].
 - 12.15. Alltech Associates. 1997. Volatile acids by Heliflex® AT-1000. Accessed 2/22/2006. [http://www.alltechweb.com/chromaccess/chromview_prn.asp?refno=1243].
 - 12.16. Agilent Technologies. 2000. Agilent J&W GC Columns: DB-XLB Columns. Accessed 3/30/2006. [<http://www.chem.agilent.com/Scripts/PDS.asp?lPage=1248>].
 - 12.17. Sigma-Aldrich. 1998. Analyzing Fatty Acids by Capillary Gas Chromatography (Supelco Bulletin 855B). Online at <http://www.sigmaaldrich.com/Graphics/Supelco/objects/4600/4509.pdf>. Accessed 3/02/2006.
 - 12.18. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
 - 12.19. SOP V1. 2006. Sampling of Volatile Organic Compounds (VOCs) in Air Samples through Use of Sorbent Tubes. Standard Operating Procedure V1. Purdue Ag Air Quality Lab.

**ANALYZING AND QUANTITATING AMINES BY ION
CHROMATOGRAPHY**

Standard Operating Procedure (SOP) V5

ANALYZING AND QUANTITATING AMINES BY ION CHROMATOGRAPHY
Standard Operating Procedure (SOP) V5

Prepared by

Bill W. Bogan, Frank M. Mitloehner (Univ. of California-Davis), and Hua Xu

Reviewed by

Albert J. Heber

Effective Date: November 13, 2007

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability.....	3
2. Summary of Method.....	3
3. Definitions of Terms and Acronyms	4
4. Health and Safety.....	4
5. Cautions	4
6. Interferences.....	6
7. Personnel Qualifications	6
8. Equipment and Supplies	7
9. Procedures	9
10. Data and Records Management	13
11. Quality Control and Quality Assurance	14
12. References.....	15

1. Scope and Applicability

- 1.1. Amines are one class of volatile organic compound (VOC) known to be emitted from some livestock operations. For example, some highly odorous manure-related compounds (e.g. trimethylamine, putrescine) are amines.
 - 1.1.1. A list of analytes which may be quantified with this method follows Section 9.4.1.
- 1.2. Ion chromatography provides a convenient method for identifying and quantitating amines collected from environmental samples (in this case, barn air). Amines are separated based on affinity towards a cation-exchange resin (which provides separation from ammonia and alkali cations), and quantitated based on conductivity (which avoids potential chromatographic interference from other, non-ionic organic compounds).
- 1.3. The method described in this SOP uses a column and mobile phase which are suited for analysis of linear and cyclic aliphatic amines.
 - 1.3.1. The method is adapted from USEPA Conditional Test Method CTM-027, and from Dionex Corporation's Product Manuals for the IonPac® CS16 and CS17 cation exchange columns.
- 1.4. Suppressed conductivity detection is used, which allows detection of most compounds down to the level of approximately 10-100 µg/L in the solution.
 - 1.4.1. This concentration is equivalent to amine concentration of 0.08-0.8 ng/L for 120-L sample. Thus, this method applies to samples containing greater than 0.1-1 ppb of any amine analyte.
- 1.5. This SOP applies to the Dionex Ion Chromatography System, consisting of the following:
 - 1.5.1. Model AS40 autosampler
 - 1.5.2. Model GP50 gradient pump
 - 1.5.3. Model CD25 conductivity detector
 - 1.5.4. Model LC20 column enclosure
 - 1.5.5. IonPac® CS17 cation exchange column
 - 1.5.6. IonPac® CG17 guard column
 - 1.5.7. Cation Self-Regenerating Suppressor (CSRS® ULTRA)
 - 1.5.8. Cation Trap Column (4-mm CTC-1)

2. Summary of Method

The chromatographic separation of individual amine components of the sample is achieved using a column packed with cation-exchange resin. The amines, which are protonated in the acidic trapping solution, display varying affinity for the resin, and are therefore separated as the mobile phase moves through the column. The column (IonPac CS17, Dionex, Sunnyvale, CA) is a hydrophilic, carboxylate-functionalized cation exchanger specifically designed for analysis of amines, and provides excellent efficiency (e.g., run time and sample throughput) and peak shape. The mobile phase consists of a mildly acidic (dilute methanesulfonic acid) solvent/water mix. Temperature control of the column is included to improve retention time reproducibility. Detection is based on conductivity increases monitored in the mobile phase as individual amines elute from the column.

3. Definitions of Terms and Acronyms

3.1.	CSRS	Cation self-regenerating suppressor
3.2.	CTC	Cation trap column
3.3.	HDPE	High-density polyethylene
3.4.	HPLC	High-performance liquid chromatography
3.5.	MΩ-cm	Megaohm-centimeter
3.6.	MSA	Methanesulfonic acid
3.7.	OSHA	Occupational Safety and Health Administration
3.8.	PAAQL	Purdue Agricultural Air Quality Laboratory
3.9.	QAPP	Quality Assurance Project Plan
3.10.	Sample batch	All samples corresponding to a single sampling event at a site (which may take place over more than one day), provided the total does not exceed ten (10) samples. More than ten samples must be divided into multiple batches for the purpose of including blanks.
3.11.	SOP	Standard operating procedure
3.12.	VOC	Volatile organic compound(s)

4. Health and Safety

- 4.1. Treat each chemical compound as a potential health hazard. Several aliphatic amines are highly flammable, and can cause severe eye damage; they are also capable of being absorbed through the skin. Some aromatic amines (e.g. certain toluidine isomers) are suspected carcinogens.
 - 4.1.1. Reduce exposure to chemicals as much as possible.
 - 4.1.1.1. Wear protective clothing (gloves, lab coats, protective eyewear) when working with chemicals.
 - 4.1.1.2. Conduct all operations involving solvents in properly-ventilated fume hoods.
 - 4.1.1.3. Keep analytical balances and other work areas clean; deal with spills promptly.
- 4.2. Methanesulfonic acid (MSA) is corrosive, and is a strong irritant. Avoid breathing the vapors while working with MSA. Always wear gloves and goggles. Prepare and handle (e.g. degas) MSA-containing mobile phases in a fume hood.
- 4.3. The laboratory will maintain a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method.
- 4.4. A reference file of material safety data sheets (MSDS) will also be maintained at PAAQL, and will be made available to all personnel involved in these analyses.

5. Cautions

- 5.1. Filter and degas all eluents (mobile phases), and filter all samples (using the filters built in to the caps of the 5-mL autosampler vials).
 - 5.1.1. Do not use bottled HPLC-grade water, since most bottled water contains an unacceptable level of ionic impurities. Use only distilled, deionized water with a resistance of 18 megohms (MΩ)/cm².
 - 5.1.2. When purging or degassing mobile phases containing solvents, do not purge or degas excessively, since this can possibly cause a volatile solvent to be “boiled” off from

- the solution, causing unintended shifts in the composition of the mobile phase. Bubble gently with helium for 5 min. Degas aqueous eluents every 24 h.
- 5.1.3. Degas and store all eluents in plastic eluent bottles which are pressurized with helium. Failure to do so can allow more-soluble atmospheric gases to enter the reservoir, and then dissolve into the mobile phase. Do not use nitrogen for this purpose, as nitrogen is soluble in solvent-containing eluents.
 - 5.2. When handling clean autosampler vials and caps, always wear disposable, powder-free PVC gloves that have been rinsed with deionized water and air-dried. Powder from gloves can be a source of contamination if it gets into samples.
 - 5.3. Failure to control the temperature of the column can result in variations in retention times, causing peaks to be misidentified.
 - 5.4. The typical operating pressure for the CS17 column or CG17 guard column is 2,000 psi, and the maximum pressure for either component is 4,000 psi. Pressures in excess of this can irreparably damage the column. Out-of range pressure will cause the IC system to beep a warning. If a different column is used in place of the CS17, consult the applicable manual for pressure limits.
 - 5.5. Always observe the following rules when switching from an eluent containing one solvent to one containing a different solvent, or when changing from a solvent-containing mobile phase to one free of solvents:
 - 5.5.1. When changing from one solvent to another, equilibrate the column for approximately 10 min with an eluent containing only 5% of the first solvent. Exchange this eluent for an eluent with 5% of the new solvent type, and equilibrate again under these conditions (approximately 10 min). Next, run a 15-min gradient, starting at 5% of the new solvent and running to the highest percentage that will be used during the new analysis protocol.
 - 5.5.2. When changing from a solvent-containing mobile phase to a solvent-free one, first equilibrate the column with between 1% and 5% of the solvent for approximately 5 min. Next, run a 10-min gradient from this solvent level to the new (0% solvent) aqueous eluent.
 - 5.5.3. The column is shipped with eluent (most often 10 mM MSA) as the storage solution; this will be specified in a test chromatogram which is included with the column. Before using a new column, equilibrate it with the desired eluent for 30 to 60 min, or until the baseline become stable.
 - 5.5.4. The column is considered to be properly equilibrated when two consecutive injections of a standard produce the same retention times.
 - 5.6. If a particular column will not be used for one week or more, prepare it for long term storage by flushing for a few minutes with the eluent. Disconnect the column, and cap both ends securely, using the plugs supplied with the column.
 - 5.7. Do not run suppressors (including the CSRS® ULTRA) at temperatures over 40°C. If the CSRS is being used, and the application requires a higher temperature, place the suppressor outside of the chromatographic jacket or oven.
 - 5.8. When using solvent containing eluents (as in this procedure), the CSRS should be used in its AutoSuppression™ External Water Mode.

- 5.8.1. Do not use the AutoRegen™ Cation Regenerant Cartridge in conjunction with the CSRS when performing these analyses, as it cannot be used with eluents containing acetonitrile. See Section 3.10 of the CS17 Product Manual.

6. Interferences

- 6.1. Ammonia will be trapped (as ammonium ions) along with the amines in these samples. If the ammonium concentration in a particular sample is high, adjust chromatographic conditions (i.e. flow rate, gradient composition) to allow for resolution of the amine peaks from that of ammonia. The risk for overlap will be highest between ammonium and short-chain aliphatic amines (e.g. methylamine, ethylamine).
- 6.2. Group I and Group II cations (e.g. lithium, sodium, potassium, magnesium, and calcium), if present in the sample, will elute at retention times similar to some common amines. Use of an in-line filter in the amine sampling procedure (SOP V3) is intended to remove any dust-entrained cations that could otherwise enter the sample. Thus, they are not expected to occur in the sample, allowing them to be used as internal standards. As a check that the in-line filter actually accomplished this, analyze samples without the cation internal standard, and compare with the internal standard alone. This will guarantee that the cations which are being used as internal standards have not been trapped with the samples.
- 6.3. Baseline shift can occur due to increasing cationic contaminant levels in the eluent as the ionic concentration of the eluent is increased over the course of the gradient analysis. Install an IonPac® Cation Trap Column (CTC-1) between the gradient pump and the injection valve. Install the CTC-1 by following the procedures in Section 3.2 of the CS17 Product Manual (“Installing the Cation Trap Column for Eluent Step change or Gradient Operation”).
- 6.4. Over time, columns can become contaminated with material that does not elute during routine analyses. Section 6 of the CS17 Product Manual (Pages 31-38) gives cleanup procedures for removing polyvalent cations (e.g transition metals), hydrophobic cations, and organic compounds from the column.

7. Personnel Qualifications

- 7.1. Personnel should be trained in the use of all applicable instrumentation, in appropriate sample-handling procedures, and in methods of data processing and analysis, before performing these analyses. It is suggested that analysts run standards of the various analytes covered in this SOP, as well as the Group I and II cations used as internal standards, to familiarize themselves with the elution order and retention times of the various analytes and standard components.
- 7.2. Each analyst must read the entire instrument manual and this SOP before working with any of the instrumentation described herein.
- 7.3. Each individual analyst must demonstrate the ability to generate acceptable results with this method.

8. Equipment and Supplies

- 8.1. Ion Chromatography (IC) system (Dionex Corporation, Sunnyvale, CA), consisting of the following components:
 - 8.1.1. Model AS40 autosampler (Fig. 1)
 - 8.1.2. Model GP50 gradient pump
 - 8.1.3. Model CD25 conductivity detector
 - 8.1.4. Model LC20 column enclosure
 - 8.1.5. IonPac® CS17 cation exchange column and CG17 guard column (or equivalent)
 - 8.1.6. Cation Self-Regenerating Suppressor (CSRS® ULTRA)
 - 8.1.7. Cation Trap Column (4-mm CTC-1)
 - 8.1.8. Software: Chromeleon Chromatography Management Systems (v. 6.7 or equivalent)
- 8.2. Methanesulfonic Acid (MSA) (>99% pure)
- 8.3. Distilled deionized (DI) water (specific resistance ≥ 18.2 M Ω -cm, which has been filtered through a 0.2-micron filter. Do not use bottled, HPLC-grade water.
- 8.4. Solvents: Ultrahigh purity HPLC/spectrophotometric solvents (acetonitrile, methanol)
- 8.5. H₂SO₄ solution for preparing standards: Add 5 mL of concentrated (98%) H₂SO₄ to 1.0 L of distilled deionized water (MilliQ water from ABE Room 101).
 - 8.5.1. Dilute 30 mL of this solution to 100 mL with distilled DI water, and use this 0.03N solution as the solvent for the aqueous calibration standards (Section 8.10.2).
- 8.6. Analytical balance: Minimum of 160-g capacity, capable of measuring to 0.0001 g
- 8.7. Volumetric flasks, Class A: Appropriate sizes with ground-glass stoppers
- 8.8. Autosampler vials: 5 mL polyvials and filter caps (Dionex Corporation P/N 038141)
- 8.9. Standards
 - 8.9.1. Stock standard and secondary dilution solutions
 - 8.9.1.1. Use the highest-purity reagents that are commercially available to make standards. Prepare stock standard solutions by accurately weighing (on an analytical balance capable of weighing 0.0001 g) about 0.0100 g of pure material. Dissolve this in ultrahigh-purity (HPLC/spectrophotometric grade) methanol and dilute to volume in a 10-mL volumetric flask.
 - 8.9.1.1.1. When compound purity is assayed to be 99% or greater, use the weight without correction to calculate the concentration of the stock standard. Otherwise, use appropriate correction factors in calculating analyte stock concentrations.
 - 8.9.1.1.2. Commercially-prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.
 - 8.9.1.2. Store stock standard solutions in amber glass bottles with PTFE-lined screw-caps. Protect from light, and maintain at -10°C (or as recommended by manufacturer).
 - 8.9.1.3. Check stock standard solutions frequently for signs of degradation or evaporation, especially just prior to using them to prepare calibration standards.
 - 8.9.1.4. Replace stock standard solutions after 6 mos.
 - 8.9.1.5. Using stock standard solutions, prepare secondary dilution standards, as needed, that contain the compounds of interest, either singly or mixed together. Prepare the secondary dilution standards at concentrations such that the aqueous calibration standards (below) will bracket the analytical system's working range.

- 8.9.1.5.1. Store secondary dilution standards with minimal headspace.
- 8.9.1.5.2. Check secondary dilution standards frequently for signs of degradation or evaporation, especially just prior to preparing aqueous calibration standards.
- 8.9.2. Aqueous calibration standards
 - 8.9.2.1. Prepare a minimum of five calibration standards at five different concentrations (e.g. a minimum of a five-point calibration curve). Prepare standards in 0.03N H₂SO₄ solution(Section 8.5.1). Each standard should contain each target compound that is intended to be detected/quantified by this method.
 - 8.9.2.1.1. The exact list of target compounds will likely be project-specific, and should be compiled before initiating a project. If new analytes of interest are identified during a project, add them to the calibration standards mixture.
 - 8.9.2.1.2. Do not report quantitative results for any compound(s) not included in the calibration standard(s).
 - 8.9.2.1.3. At least one of the calibration standards should correspond to a concentration at or below that necessary to meet the data quality objectives of the project.
 - 8.9.2.1.4. The remaining standards should correspond to the range of concentrations found in actual samples but should not exceed the working range of the IC.
 - 8.9.2.1.5. Note the following precautions when preparing aqueous calibration standards from secondary dilution standards:
 - 8.9.2.1.5.1. Do not inject methanolic standards into water at levels higher than 20 µL standard into 100 mL of water.
 - 8.9.2.1.5.2. Use a 25-µL Hamilton 702N microsyringe or equivalent. Variations in needle geometry in other syringes will adversely affect the ability to deliver reproducible volumes of standard. Never use pipettes to dilute or transfer samples or aqueous standards when diluting VOC standards.
 - 8.9.2.1.5.3. Fill the sample syringe from the standard solution contained in the expanded area of the flask (do not use solution from the neck of the flask).
 - 8.9.2.1.5.4. Rapidly inject the secondary dilution standard into the filled volumetric flask, and remove the needle as quickly as possible after injection.
 - 8.9.2.1.5.5. Mix diluted standards by inverting the flask three times only.
 - 8.9.2.1.6. Spike each 1-mL aliquot of calibration standard with 10 µL of the internal standard solution (Section 8.8.3) prior to analysis.
 - 8.9.2.2. Prepare the calibration standard weekly and store at 4°C.
- 8.9.3. Internal standards
 - 8.9.3.1. Choose internal standard(s) such that most of the analytes of interest in a chromatogram will have retention times between 80% and 120% of the retention time of one (or more) of the internal standards. Suitable internal standards for this method include Group I and II cations, specifically Li, Ca, K, and Mg. Verify that none of these are already present in the samples (Section 6.2.1).
 - 8.9.3.2. Dissolve LiCl (0.6108 g), KCl (0.1907 g), MgCl₂•6H₂O (0.8365 g), and CaCl₂•2H₂O (0.3668 g) in a small volume of deionized, distilled water. Transfer to a 50-mL volumetric flask and dilute to volume.
 - 8.9.3.3. Spike each 1-mL sample extract with 10 µL of the internal standard solution, resulting in a concentration of 20 ng/µL of each internal standard cation.

- 8.9.3.4. Area counts of the internal standard peaks should be between 50-200% of the areas of the target compounds which are present in the midpoints of the calibration curves for those compounds.
- 8.9.3.5. Store internal standard mixtures at 4°C (refrigerated, not frozen) when not in use.
- 8.9.3.6. Prepare fresh internal standard mixtures once a year.
- 8.9.3.7. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

9. Procedures

9.1. Pre-analysis sample handling

- 9.1.1. Keep samples refrigerated (not frozen) at 4°C (39°F) prior to analysis.
- 9.1.2. Allow samples to slowly warm to lab temperature just before initiating analysis.
- 9.1.3. Always analyze samples within 14 days after their collection in the field.

9.2. Sample preparation

9.2.1. Bubbler solutions:

- 9.2.1.1. Pour the solutions from bubblers 1 and 2 from their HDPE sampling bottles into separate 50-mL volumetric flasks.
- 9.2.1.2. Rinse out the interior walls of the bottles several times with approximately 5-mL portions of deionized water; add each rinse to the applicable 50-mL volumetric flask until the total volume reaches the mark.
- 9.2.1.3. This process takes the 0.1N H₂SO₄ impinger solution to an approximately 0.03N H₂SO₄ solution, an acidity level which is compatible with the CS17 column.
 - 9.2.1.3.1. If 0.1 N H₂SO₄ was used to rinse the impingers (Step 7.4.3 of SOP V3), then it will be necessary to dilute the sample by a factor of three (3) to five (5) to reach the 0.02–0.03 N level that is compatible with the column. Use a suitable dilution factor in subsequent calculations.
- 9.2.1.4. Add 500 µL of internal standard solution to the diluted samples in 50-mL volumetric flasks.
- 9.2.1.5. Fill autosampler vials and use insertion tool to push the cap into the vial (Fig. 2); then use the other end of insertion tool to level off the caps. Fill vials to the shoulder or below. Do not fill to the top. Cap all vials tightly.
- 9.2.1.6. Load the vials into the AS40 Autosampler tray. Load the tray into the autosampler, and close the compartment door.
- 9.2.1.7. Turn the autosampler power on press “run”.

9.3. Sample analysis

9.3.1. Ion chromatography conditions

- 9.3.1.1. Proper system configuration (flow rates, sample injection volumes, configuration of pumps, etc.) is given in Section 2 (Pages 7-8) and Section 4.1 of the CS17 Manual. Several parameters will vary, depending on whether a 2-mm diameter column (cation capacity = 325 µeq) or a 4-mm column (1300 µeq) is used.
- 9.3.1.2. Suitable gradient profiles for analysis of amines are given in Section 5 of the CS17 Product Manual. In general, this SOP uses a mobile phase consisting of 3-6 mM MSA, at a flow rate of 1.0-1.5 mL/min.

- 9.3.1.3. Operating conditions may need to be adjusted to allow for resolution of the amine peaks from those of the various cations in the internal standard.
 - 9.3.1.3.1. The final gradient program must achieve separation of all analytes and internal standard components in the highest-concentration calibration standard such that the intersection between any two adjacent peaks (the “valley” on the chromatographic profile) is no more than 25% of the mean height of the two peaks themselves. Resolution all the way to baseline is not required.
- 9.3.1.4. Program the GP50 Gradient Pump to deliver the above gradient profile. See Section 3 of the GP50 Operator’s Manual for further detail.
- 9.3.1.5. Make sure that the CD25 Conductivity Detector is operating properly, and is programmed for the correct method. See Section 3 of the CD25 Operator’s Manual for further detail.
- 9.3.2. Inclusion of standards in analytical runs
 - 9.3.2.1. Bracket each sample batch with a set of the five standards for the standard curve, run in duplicate. Arrange the standards in each set from low to high concentration (Section 11.3.3), and include a solvent blank after each highest-concentration standard (Section 11.3.4).
 - 9.3.2.2. Use a quadratic regression to generate the standard curve.
 - 9.3.2.2.1. In certain cases (those where all concentrations are in the range of 10-100 ppb), linear regression can also be used for some amines.
 - 9.3.2.2.2. Do not include the origin for the purposes of generating the standard curve.
- 9.3.3. Flush the CTC-1 at the end of each day of operation to remove any impurities that may have accumulated on it.
 - 9.3.3.1. At the end of the day, disconnect the CTC outlet, and connect a waste line to it.
 - 9.3.3.2. Using a 10x concentration of the strongest eluent required in the sample analysis, run 30 mL through the CTC at a flow rate of 2 mL/min.
 - 9.3.3.3. At the beginning of the next analysis day, flush the CTC with 30 mL of a 1x solution of the strongest eluent used in the gradient program.
 - 9.3.3.4. Reconnect the CTC to the eluent line.
- 9.3.4. Calculations
 - 9.3.4.1. Export or cut and paste the data from the IC quantitation to a Microsoft Excel spreadsheet reserved for amine concentration calculations.
 - 9.3.4.2. Determine total volume of air sampled by multiplying average flow rate of the sampling pump (Section 8.3.8 of SOP V3), which is recorded in the electronic field notes, by the total sampling time. Average flow rate of the pump is the average value between the beginning and ending flow rates, provided that the two did not differ by more than 10%.
 - 9.3.4.2.1. If the difference between the initial and final flow rates is greater than 10%, invalidate the sample.
 - 9.3.4.3. Correct this sample volume to standard conditions (20 °C, 760 mm Hg) using the following equation. Express $V_m(\text{std})$ in L (One $\text{m}^3 = 1000 \text{ L}$).

$$V_{m(\text{std})} = V_m Y \left(\frac{T_{\text{std}}}{T_m} \right) \left[\frac{P_{\text{bar}} + \frac{\Delta H}{13.6}}{P_{\text{std}}} \right]$$

Where:

$V_{m(\text{std})}$ = Volume of gas sample, corrected to standard conditions

V_m = Volume of gas sample

T_{std} = Standard absolute temperature, 293 K

T_m = Absolute average temperature during sampling, K

P_{bar} = Barometric pressure at the sampling site, mm Hg

P_{std} = Standard absolute pressure, 760 mm Hg

ΔH = Average pressure differential across the orifice meter, mm H₂O

13.6 = Specific gravity of mercury

9.3.4.4. Determine the concentration of each amine in the diluted impinger solution by application of the ion chromatography (IC) calibration equation. Express this in milligrams of each amine compound per liter of solution.

9.3.4.5. Calculate the volume of each individual amine analyte present in the sample:

$$V_a = \frac{(N)(0.1)(24.04)(0.001)}{(FW_a)}$$

Where:

V_a = Volume of individual amine gas in the sample of gas from the source

N = Mean amine concentration (mg/L) between the two impinger solutions ((Impinger 1 Conc. + Impinger 2 Conc.)/2)

0.1 = Conversion factor, assuming sample in each of the two impingers was diluted to 50 mL (0.05 L)

24.04 = Liters of ideal gas per mole of substance

0.001 = Factor to convert mg/L to g/L

FW_a = Formula weight of amine analyte (g/mole). Formula weights of various possible target analytes for this SOP include those listed in Table 1.

9.3.4.4. 9.3.4.6. Calculate the ppmV (C_a) of each amine analyte present in the gas sample:

$$C_a = \frac{V_{a, L}}{V_{m(\text{std}), L}} \times 10^6$$

9.4. Troubleshooting

9.4.1. Section 6 of the CS17 Product Manual provides a troubleshooting section for issues relating to contamination, elevated back pressure and/or background conductivity, background noise and/or unstable baselines, low retention times, poor peak shape and/or resolution, and spurious peaks. If any of these problems are encountered, consult Section 6 of the manual.

Table 1. Target compounds analyzed using this method.

Analyte	Formula weight, g/mol
Hydrazine (N ₂ H ₄)	32.00
2-Butylamine	73.14
<i>n</i> -Butylamine	73.14
<i>t</i> -Butylamine	73.14
Diethylamine	73.14
Dimethylamine	45.08
Ethylamine	45.08
Ethylene diamine	60.10
Isoamylamine (3-methylbutylamine)	87.17
Isobutylamine (2-methylpropylamine)	73.10
Isopropylamine	59.11
Methylamine	31.06
1-Pentanamine (amylamine)	87.15
Propylamine	59.11
Trimethylamine	59.11
Triethylamine	101.19
Putrescine (1,4-diaminobutane)	88.15
Aminopyridine	94.12
Methylpyrazine	94.12
Dimethylpyrazine	108.14
Trimethylpyrazine	122.16
Tetramethylpyrazine	136.18

- 9.4.1.1. High system pressure can be due to blockages in column bed supports, or other hardware components. Isolate the blockage and clean or replace the part.
- 9.4.1.2. High background readings and/or high “noise” levels in the background can be due to a malfunctioning or contaminated CSRS, an air bubble trapped in the CSRS, contaminants in the mobile phase, contaminants in the column, or a defective proportioning valve. Check each of these possibilities, and address with guidance from the manual. If air bubbles are found, ensure that the eluent is being properly degassed, and that the reservoir is remaining under positive pressure with helium
- 9.4.1.3. Poor resolution can be due to column overloading, excessively low sample pH, contaminants in the system and/or column, or a damaged column. Dilute sample, check sample pH against the appropriate range for the CS17, or replace the column and/or its bed supports, as needed to isolate and fix the problem.
- 9.4.1.4. Poor quantitation of divalent cations (the internal standard) is most likely due to contamination in the sample loop, CSRS, or detector cell. Clean or replace these components as necessary.
- 9.4.2. Troubleshooting for the CD25 Conductivity Detector is addressed in Section 4 of its Operator’s Manual.

- 9.4.2.1. If the detector shows no response, check the following:
 - 9.4.2.1.1. Detector cell may be turned off.
 - 9.4.2.1.2. Detector may not be receiving flow (pump turned off or not working).
 - 9.4.2.1.3. Selected output range may be too high or too low, or no output range may be selected.
- 9.4.2.2. If the detector response is present but very low, check the following:
 - 9.4.2.2.1. Selected output range is too high.
 - 9.4.2.2.2. Sample may be too dilute.
 - 9.4.2.2.3. Detector cell may require calibration.
- 9.4.2.3. The following can cause unstable or noisy baselines:
 - 9.4.2.3.1. Leaks in the system. Check all connections for possible leaks.
 - 9.4.2.3.2. Rapid or significant fluctuations in temperature. Check the room housing the system for temperature variations.
 - 9.4.2.3.3. Pump may be out of prime. Re-prime as necessary.
 - 9.4.2.3.4. Air trapped in the detector cell
- 9.4.3. Troubleshooting for the GP50 Gradient Pump is addressed in Section 4 of its Operator's Manual.
 - 9.4.3.1. If operating pressure fluctuates, re-prime the pump, check that sufficient mobile phase is available, and that the mobile phase is fully degassed. Check for blockages to inlet filters and lines, and check for dirty or defective piston seals and check valves; clean or replace as necessary.
 - 9.4.3.2. If pump will not stay on, check electrical connections. Verify that pressure limits (high or low) have not been exceeded. If high pressure limits have been exceeded, See Section 9.4.1.1 above.
 - 9.4.3.3. Leaks most commonly occur at the piston seal, proportioning valve, priming valve, and pressure transducer.
- 9.4.4. If either the blanks (Section 11.4), the quality assurance audits (Section 11.6), or the quality control check (Section 11.7) fail to meet their acceptance criteria, check the IC system first (as above) to make sure every parameter is correct, then do the calibration again. If the problem cannot be located and solved using the above information, technical support from the company may be needed.

10. Data and Records Management

- 10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
- 10.2. Manage all data according to SOP B5.
- 10.3. Document all data and information on field data sheets, and within site logbooks with permanent ink, or in electronic field notes.
- 10.4. Overstrike all errors in writing with a single line. Initial and date all such corrections.

11. Quality Control and Quality Assurance

11.1. Reagent purity

11.1.1. Use reagent-grade chemicals. Unless otherwise indicated, all reagents will conform to the specifications, if any, of the Committee on Analytical Reagents of the American Chemical Society. If any other grade of reagent is to be used, ascertain first ascertained that it is of sufficiently high purity to be used without affecting the accuracy of the determination.

11.1.2. Use only ultrahigh purity, HPLC/spectrophotometric grade or equivalent solvents, and store these away from other solvents.

11.2. Cleaning and processing of glassware

11.2.1. Rinse glassware as soon as possible after use with the last solvent used in it.

11.2.2. Wash in hot water with detergent, rinse with tap water and distilled water. Drain dry.

11.2.3. Heat at 400°C in a muffle furnace for 15-30 min.

11.2.3.1. Solvent rinses with acetone and pesticide quality hexane may be substituted for the muffle furnace heating.

11.2.3.2. Volumetric glassware should not be heated in a muffle furnace.

11.2.4. When cool, glassware should be sealed and stored (inverted or capped with aluminum foil) in a clean (i.e. dust-free) environment.

11.3. Calibration

11.3.1. Prepare a calibration curve each analysis day using a minimum of five standards that bracket the expected range of sample concentrations. One of these five should be at or below the analyte concentration which corresponds to the minimum detection limit of the project, as stated in the QAPP.

11.3.2. Prepare calibration standards in filtered, degassed 0.03N H₂SO₄, the same concentration of acid as in the diluted samples.

11.3.3. Analyze the calibration standards from lowest concentration to highest concentration.

11.3.4. Verify that the system is clean (i.e. that there is no carryover) by analyzing a solvent blank after the highest-concentration calibration standard.

11.3.5. Base calibration curves on peak area, rather than peak height.

11.3.6. To be acceptable, the R² value for the calibration curve must be ≥0.98.

11.3.7. Keep a control chart (SOP Q1) of calibration curve results, so that they may be compared over time to assess possible detector drift or other biases/inaccuracies that may manifest themselves over time. If the average peak area per unit concentration of any given analyte drifts from its baseline value (that at the beginning of the study) by more than ±10%, troubleshoot the system (Section 9.4) to identify and rectify the problem.

11.4. Blanks

11.4.1. A minimum of one method blank and one trip blank (SOP V3) will be included with each batch of samples, and must be processed and analyzed along with those samples.

11.4.2. To be acceptable, no target analyte may be present above its detection level in either the field or trip blank.

11.4.3. Analyze column blank(s) whenever the analysis of a method blank indicates that cross-contamination may be occurring.

11.5. Reproducibility

11.5.1. Run a minimum of 20% of the samples in duplicate to assess reproducibility. Report results for these samples as mean concentrations of the duplicate analyses, with standard deviation. If the standard deviation between duplicate analyses exceeds 10%, prepare and re-analyze two new vials of the same sample with the next run.

11.6. NIST-traceable aqueous standards containing known amounts of amine analytes are available from various sources. For example, 1000-ppm standards of dimethylamine and trimethylamine are available from Alltech Associates, Deerfield, IL. Analyze these standards at the beginning of the study, and then use them for quality assurance audits of the analytical process, which are to be conducted quarterly at minimum.

11.6.1. If the analytical results are not within $\pm 2\%$ of the known concentration, check operating parameters (Section 9.3.1), troubleshoot the IC system (Section 9.4) and repeat the analysis. If necessary, recheck all calibration standards against the control chart (Section 11.3.7). Contact manufacturer technical support if the problem cannot be identified.

11.7. Conduct quality control checks by analyzing a solution that contains at least two of the amine analytes, at concentration(s) which are in the range of the calibration standards, but that has been prepared independently using different source(s) of amines salt than those used to prepare the calibration standards.

11.7.1. Perform this check monthly.

11.7.2. Perform this check whenever a new set of calibration standards is prepared.

12. References

- 12.1. USEPA. 1997. Conditional Test Method CTM-027 – Procedure for Collection and Analysis of Ammonia in Stationary Sources. Online at www.epa.gov/ttn/emc/ctm/ctm-027.pdf. Accessed 3/3/2006.
- 12.2. Dionex Corporation. 2003. Product Manual – IonPac CG17 Guard Column/IonPac CS17 Analytical Column. Online at http://www1.dionex.com/en-us/webdocs/4392_31877-03_CS17_V18.pdf. Accessed 4/6/2006.
- 12.3. Dionex Corporation. 2001. Determination of Inorganic Cations and Ammonium in Environmental Waters by Ion Chromatography Using the IonPac® CS16 Column. Online at http://www1.dionex.com/en-us/webdocs/4211_AN141_V15.pdf. Accessed 3/14/2006.
- 12.4. Dionex Corporation. 2001. GP50 Gradient Pump Operator's Manual (Rev. 03). Online at http://www1.dionex.com/en-us/webdocs/4533_31377-03_V12.pdf. Accessed 3/17/2006.
- 12.5. Dionex Corporation. 2000. CD25 Conductivity Detector Operator's Manual (Rev. 01). Online at http://www1.dionex.com/en-us/webdocs/4518_31687-01.pdf. Accessed 3/17/2006.
- 12.6. Dionex Corporation. 1997. IonPac® CS17 Cation Exchange Column – Product Data Sheet. Online at http://www1.dionex.com/en-us/webdocs/4259_CS17_V18.pdf. Accessed 4/6/2006.
- 12.7. Dionex Corporation. 1994. AS40 Automated Sampler – Operator's Manual. Document #034970, Revision 2.
- 12.8. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.

- 12.9. SOP Q1. 2006. Use of Control Charts for Performance Monitoring of Gas Analyzers and Analytical Instruments. Standard Operating Procedure Q1. Purdue Ag Air Quality Lab.
- 12.10. SOP V3. 2006. Collection of Amines in Air Samples Through Use of Sulfuric Acid-Containing Impingers. Standard Operating Procedure V3. Purdue Ag Air Quality Lab.

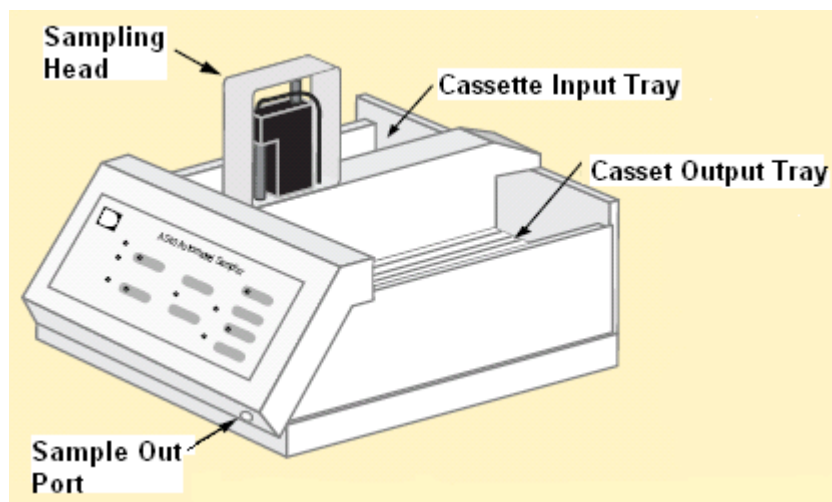


Figure1. Diagram of the Dionex AS 40 Autosampler

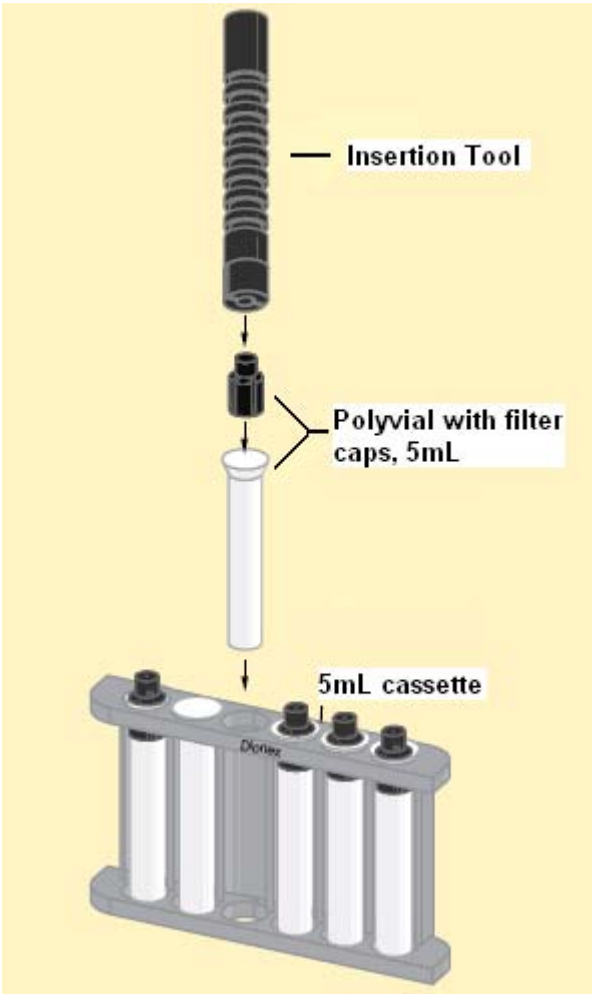


Figure 2. Autosampler vials, cassette and insertion tool used for inserting the filter caps.

**GC/MS ANALYSIS OF PHENOLS AND VOLATILE FATTY ACIDS
(VFAs)**

COLLECTED IN SAMPLING CANISTERS

Standard Operating Procedure (SOP) V6

**GC/MS ANALYSIS OF PHENOLS AND VOLATILE FATTY ACIDS (VFAs)
COLLECTED IN SAMPLING CANISTERS
Standard Operating Procedure (SOP) V6**

Prepared by

Bill W. Bogan and Hua Xu

Reviewed by

Albert J. Heber

Effective Date: November 6, 2006

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	4
3. Definitions	4
4. Health and Safety	6
5. Cautions	6
6. Interferences	6
7. Personnel Qualifications.....	8
8. Equipment and Supplies	8
9. Procedures	10
10. Data and Records Management	17
11. Quality Control and Quality Assurance	17
12. References.....	22

1. Scope and Applicability

- 1.1. This SOP describes a gas chromatography/mass spectrometry (GC/MS)-based method for detecting and quantitating volatile organic compounds (VOCs) in ambient air at livestock facilities.
- 1.1.1. The specific classes of VOC covered under this SOP are phenols and volatile fatty acids (VFAs), including the specific analytes listed in Table 1 below.

Table 1. Target analytes.

VFAs	Phenols
Acetic acid	Phenol
Propionic acid	p-cresol
Butyric acid	o-cresol
Isobutyric acid	m-cresol
Valeric acid	p-ethylphenol
Isovaleric acid	m-ethylphenol
Hexanoic acid	o-ethylphenol
Iso-caproic acid	Phenol
2-methylpentanoic acid	p-cresol
Heptanoic acid	o-cresol
Octanoic acid	m-cresol
Nonanoic acid	
Decanoic acid	
Undecanoic acid	
Dodecanoic acid	
Tridecanoic acid	
Tetradecanoic acid	
Benzoic acid	
Phenyl acetic acid	
3-phenylpropanoic acid	

- 1.2. This method analyzes VOCs collected using sampling canisters (SOP V2).
- 1.3. The minimum detection limit is approximately 0.5 parts per billion (ppbv) concentration in the air stream in question.
- 1.3.1. The detection limit will be somewhat dependent upon the degree of VOC enrichment achieved by concentrating the sample. It may be necessary to concentrate up to one liter of sample per injection to reach this detection limit for some analytes.
- 1.4. The methodology described in this SOP is derived from the following sources:
- 1.4.1. USEPA Method TO-14A – “Determination of Volatile Organic Compounds (VOCs) in Ambient Air Using Specially Prepared Canisters with Subsequent Analysis by Gas Chromatography”
- 1.4.2. USEPA Method TO-15 – “Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)”

- 1.4.3. USEPA Method 8260B – “Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)”
- 1.4.4. USEPA Method 8270C – “Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)”
- 1.4.5. California EPA Air Resources Board SOP MLD 059 – “Determination of Oxygenated Hydrocarbons (OHCs) in Ambient Air by Capillary Column Gas Chromatography/Mass Spectrometry”.

2. Summary of Method

Sampling canisters are used to collect VOC analytes according to SOP V2. Canisters are leak-checked after arrival in the laboratory, and are pressurized (if necessary) with humidified ultra-high-purity nitrogen. An aliquot of sample (up to 1,000 mL) is withdrawn from the canister, and introduced into a cryogenic concentrator. Water content of the sample can be reduced at this point, if necessary, by dry purging the concentrator with helium, while retaining target compounds. The analytes are thermally desorbed from the concentrator into a cryofocusing unit, which is coupled with a gas chromatograph (GC) inlet. Analytes are refocused on the trap of the cryofocusing unit, and concentrated to a very small volume. Rapid desorption of the trap follows, with transferring of the sample into the GC (Agilent Model 6890), which separates individual analytes by using a suitable GC column. Compound identification is accomplished by comparing their GC retention times and mass spectra (determined with an Agilent MSD 5975 Mass Spectrometer) with those of authentic standards. Quantitation of each compound is done by comparing the response (preferably peak area of its primary characteristic ion, but peak height may also be used) with those in a five-point calibration curve (the midpoint of which is verified by a daily calibration check).

3. Definitions

- 3.1. **Thermal Desorption:** The use of heat and a flow of inert (carrier) gas to extract volatiles from a solid matrix directly into the carrier gas and transfer them to a downstream system element. No solvent is required.
- 3.2. **Focusing Tube (Focusing Trap):** Narrow (typically < 3 mm I.D.) tube containing a small bed of sorbent, which is maintained near or below ambient temperature and used to refocus and concentrate analytes too dilute to be analyzed. Once all the VOCs have been transferred from the sorbent tube to the focusing tube, the focusing tube is heated very rapidly to transfer the analytes into the capillary GC analytical column in a narrow band of vapor (equivalent to a small injection volume), which is compatible with high resolution capillary gas chromatography.
- 3.3. **Cryofocusing:** Use of coolant (e.g., liquid nitrogen, liquid argon, or liquid CO₂) to lower the temperature of the focusing trap and promote more rapid and complete sorption of analytes on the trap.
- 3.4. **Dynamic Calibration:** Calibration of an analytical system using calibration gas standard concentrations in a form identical or very similar to the samples to be analyzed and by introducing such standards into the inlet of the sampling or analytical system from a manifold through which the gas standards are flowing.

- 3.5. **Dynamic Dilution:** A means of preparing calibration mixtures in which standard gas(es) from pressurized cylinders are continuously blended with humidified zero air in a manifold so that a flowing stream of calibration mixture is available at the inlet of the analytical system.
- 3.6. **Relative Retention Time (RRT):** The ratio of the retention time of an individual analyte over the retention time of the internal standard with which the analyte is being compared for purposes of identification. RRT is a unitless quantity, as the numerator and denominator time units cancel out. For identification of the analyte peak to be valid, RRT of the sample component is within ± 0.06 RRT units of RRT of the standard component (EPA Method 8260B).
- 3.7. **Response Factor (RF):** Instrument sensitivity towards a particular compound. The relationship between instrument output (e.g. peak height or integrated area) and analyte concentration.
- 3.8. **Relative Response Factor (RRF):** The comparison of the Response Factor of an analyte (i.e. a calibration standard, target compound, or surrogate) to that of an internal standard.
- 3.9. **MS-SCAN:** Mode of operation of a GC/MS in which all mass ions over a given mass range are swept over a given period of time.
- 3.10. **MS-SIM:** Mode of operation of a GC/MS in which only a single mass ion (or a selected number of discrete mass ions) are monitored.
- 3.11. **Total Ion Chromatogram (TIC):** Chromatogram produced from a mass spectrometer detector operating in full scan (MS-SCAN) mode.
- 3.12. **Extracted Ion Current Profile (EICP):** A plot showing the abundance of an ion of a particular mass, plotted as a function of retention time or scan number.
- 3.13. **Primary Characteristic Ion:** The particular ion, defined by USEPA or other methods, which provides the best results in terms of quantitating a particular compound.
- 3.14. **Target Compound:** A compound designated by the Statement of Work for analysis (qualitative or quantitative)
- 3.15. **Tuning Standard:** A standard used to (a) verify that the MS system is operating properly and (b) guard against reactive sites in the system (especially the injection ports, lines, and the first segments of the column) that might cause chemical breakdown or degradation of the sample
- 3.16. **Method Blank:** A blank sample (i.e. from a cylinder of zero air) that is treated exactly as a sample, and is processed simultaneously with other samples in the analysis batch through all steps of the analytical procedure. This is done to determine if method analytes or other potential interferences are present in the lab environment, reagents, or apparatus, and/or to check for sample carryover.
- 3.17. **Calibration Check Compounds (CCC):** A group of compounds used as a standard to evaluate the calibration from the standpoint of system integrity. Highly variable results with the CCCs may indicate system leaks or reactive sites on the column.
- 3.18. **System Performance Check Compounds (SPCC):** SPCCs are compounds that are known to have very low RFs (0.1-0.2), and that tend to decrease rapidly in response as the chromatographic system (or the standard material) begins to deteriorate. They are usually the first to show poor performance, and are therefore used to ensure that the system is performing adequately.

- 3.19. **Replicate Precision:** The precision determined from two canisters filled from the same air mass over the same time period. It is the absolute value of the difference between the analyses of the two canisters, divided by their average value, and expressed as a percentage.
- 3.20. **Duplicate Precision:** The precision determined from the analysis of two samples taken from the same canister. It is the absolute value of the difference between the analyses of the two samples, divided by their average value, and expressed as a percentage.

4. Health and Safety

- 4.1. Follow appropriate safety measures when working with pressurized gas cylinders. See SOP G2 for more detail.
- 4.2. Each chemical compound should be treated as a potential health hazard, and exposure to these chemicals will be reduced to the lowest possible level.
- 4.2.1. Wear protective clothing (gloves, lab coats, protective eyewear) when working with chemicals.
- 4.2.2. Conduct all operations involving solvents in properly-ventilated chemical fume hoods.
- 4.2.3. Keep analytical balances and other work areas clean; deal with spills promptly.
- 4.3. Maintain a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method.
- 4.4. Also maintain a reference file of material safety data sheets (MSDSs), which will be made available to all personnel involved in these analyses.

5. Cautions

- 5.1. Contact between the capillary column and GC oven wall affects chromatographic performance and column life. Do not allow column to touch the oven walls.
- 5.2. Carrier gas should be scrubbed to remove traces of oxygen and scrubbers should be changed regularly. If the Agilent “big universal trap” (Section 8.9.1) is used to purify helium, it should be changed after every 13th helium cylinder.
- 5.3. The use of a guard column between the injection port and the analytical column may help prolong analytical column performance. Use guard columns, either as part of a pre-fabricated column or separate (Section 8.2).
- 5.4. The period between sampling and analysis should never exceed 30 d

6. Interferences

- 6.1. Several loss mechanisms exist for analytes collected into sampling canisters.
- 6.1.1. Losses due to physical adsorption occur as equilibrium is reached between the condensed and gas phases of an analyte. These are generally considered short-term losses, (i.e., losses occurring over minutes to hours). These may be more significant with low-humidity samples.

- 6.1.2. Conversely, if water condenses in the canister, analytes can dissolve in this water, reducing recovery. This can be particularly significant if high-humidity samples are pressurized during dilution.
- 6.1.3. Losses due to chemical reactions of the VOC analytes with co-collected ozone or other reactive, unstable gas-phase species also account for some short-term losses.
- 6.1.4. Chemical reactions between VOCs and other, more stable substances inside the canister are generally assumed to cause the gradual decrease of concentration over time (i.e., long-term losses over days to weeks).
- 6.1.5. Loss mechanisms based on aqueous hydrolysis also exist.
- 6.1.6. Biodegradation of analytes can also occur over time.
- 6.1.7. Under conditions of normal usage for sampling ambient air, most VOCs can be recovered from canisters near their original concentrations after storage times of up to 30 d (Method TO-15).
- 6.2. Impurities in the canister pressurizing gas, calibration dilution gas (if applicable) and carrier gas, organic compounds out-gassing from the system components ahead of the trap, and solvent vapors in the laboratory can all result in contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running humidified zero air blanks.
 - 6.2.1. Never use non-chromatographic grade stainless steel tubing, non-PTFE thread sealants, or flow controllers with Buna-N rubber components.
- 6.3. Use of other than inert materials (deactivated fused silica, glass lined tubing, glass, quartz and PTFE) in the sample flow path can result in degradation of some analytes.
- 6.4. Very volatile compounds can display peak broadening and co-elution with other species if the compounds are not delivered to the GC column in a small volume of carrier gas. Cryofocusing of the sample mitigates this problem.
- 6.5. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered, follow with a suitable blank to check for cross-contamination.
- 6.6. When sample components are not resolved chromatographically, the result will be a mass spectrum containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s), or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.
- 6.7. When analytes coelute (i.e., only one chromatographic peak is apparent), the criteria for identification may be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound. In these cases, examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra and in qualitative identification of compounds.
- 6.8. Compounds that are too labile or reactive for conventional GC analysis are not suitable for this method.
- 6.9. Several phenolic compounds outside the scope of this SOP (e.g. 4-nitrophenol, 2,4-dinitrophenol, 4,6-dinitro-2-methylphenol, and 4-chloro-3-methylphenol) can be subject to erratic chromatographic behavior when analyzed by this method; this is especially true if the GC system is contaminated with high-boiling materials (high-molecular-weight, nonvolatile compounds).

- 6.9.1. It is therefore possible that phenols relevant to this SOP may also display this behavior; care should thus be taken to keep the system free of contamination of this nature. Use phenol-specific System Performance Check Compounds (SPCCs, Section 9.3.3) to guard against this.

7. Personnel Qualifications

- 7.1. Personnel should be trained in the use of all applicable instrumentation, and in appropriate sample-handling procedures, before performing the activities in this SOP.
- 7.2. Each analyst must read and understand the applicable equipment manuals and this SOP before performing these activities.
- 7.3. Each individual analyst must demonstrate the ability to generate acceptable results with this method (Section 11.12).

8. Equipment and Supplies

- 8.1. Gas chromatograph: Agilent GC6890 System
 - 8.1.1. Six-Port Gas Chromatographic Valve: Seiscor Model VIII, Seismograph Service Corp., Tulsa, OK (or equivalent)
 - 8.1.2. This GC meets the required capabilities detailed in USEPA Method 8270C, USEPA Compendium Method TO-15, and California EPA ARB SOP MLD 059.
- 8.2. Column: Columns that are suitable for use under this SOP are the following:
 - 8.2.1. Heliflex[®] AT-1[™]ms low-bleed capillary GC column (Part #935110): Alltech Associates, Deerfield, IL. Should include a 5-m “leader” span of column, which is not coated with stationary phase, and therefore functions as a guard column.
 - 8.2.2. DB-XLB capillary GC column, 30m × 0.25mm × 0.25μm (Part #122-1232G): Agilent Technologies, Palo Alto, CA. Should include a 10-m “DuraGuard” span of column, which functions as a guard column.
 - 8.2.3. An equivalent column (or column/guard column combination) may be used if equal performance can be achieved.
- 8.3. Mass spectrometer: Agilent 5975 Inert MSD System
 - 8.3.1. This MS meets the required capabilities detailed in USEPA Method 8270C, USEPA Compendium Method TO-15, and California EPA ARB SOP MLD 059.
 - 8.3.2. Perfluorotributylamine (PFTBA) is used as the tuning compound for electron impact tuning.
- 8.4. GC/MS Interface: Capillary-direct into the MS source
 - 8.4.1. Meets the required capabilities detailed in Methods 8270C and TO-15
- 8.5. Data Analysis System: A computer system that will allow continuous acquisition and storage of all mass spectra. Vendor (Agilent)’s software allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number (Extracted Ion Current Profile (EICP)). Vendor’s software also allows integrating the abundances in any EICP between specified time or scan-number limits.
 - 8.5.1. Use the most recent version of the EPA/NIST Mass Spectral Library.

- 8.6. A dynamic calibration system (Fig. 1) consists of the following components:
 - 8.6.1. Calibration manifold: Glass manifold (1.25 cm I.D. × 66 cm) with sampling ports and internal baffles for flow disturbance to ensure proper mixing.
 - 8.6.2. Humidifier: 500-mL impinger flask containing HPLC-grade deionized water.
 - 8.6.3. Electronic mass flow controllers: One 0 to 5 L/min and one 0 to 50 mL/min
 - 8.6.4. Teflon filter(s): 47 mm, for particulate control
- 8.7. Cryogenic/Adsorbent Concentrator, with the following components:
 - 8.7.1. Adsorbent trap, ca. 1/8" (700 µL), packed with 60/80 Carbopack B, Carbopack C, and Carboxen 1000.
 - 8.7.2. Multi-position Stream Selector Valve
 - 8.7.3. Low-pressure regulator with Teflon-lined diaphragm
 - 8.7.4. Mass flow controller, rated at 100 cm³/min at full scale
 - 8.7.5. Fixed-volume sample loop for addition of internal standard, when required
- 8.8. Tubing for connecting canisters to the Cryogenic/Adsorbent Concentrator
 - 8.8.1. The following types and sizes are appropriate: 1/8" Teflon, 1/16" stainless steel, or 1/16" glass-lined tubing.
- 8.9. Helium cylinders - 99.999% or higher purity He as the GC carrier gas
 - 8.9.1. Scrubber: Use the Agilent "big universal trap" (Part # RMSH-2), or equivalent, to purify the helium carrier gas.
- 8.10. Nitrogen cylinders - 99.999% or higher purity N₂ as the diluent gas for pressurizing canisters (Section 9.4.1)
- 8.11. Liquid N₂ for cooling of the GERSTEL Cooled Inlet System (CIS)
- 8.12. Calibration System
 - 8.12.1. Calibration Manifold: Stainless steel, glass, or high purity quartz manifold with sampling ports and internal baffles for flow disturbance to ensure proper mixing. The manifold should be heated to ~ 50°C.
 - 8.12.2. Humidifier: 500-mL impinger flask containing HPLC grade deionized water.
 - 8.12.3. Electronic Mass Flow Controllers: 0 to 5 L/min and 0 to 100 mL/min units
 - 8.12.4. Teflon Filters (47-mm) for particulate collection
- 8.13. Calibration standards
 - 8.13.1. Calibration standards must include each target analyte in a particular project. This information will be included in the QAPP and/or Statement of Work.
 - 8.13.2. Prepare standards in humidified zero air, using one of the methods in Section 9.1.
 - 8.13.3. Prepare working standard mix at a concentration (each analyte) of 10 ppmv.
 - 8.13.3.1. Consult the QAPP or statement of work to determine the range for each analyte.
 - 8.13.3.2. When reagent purity is 96% or greater, it is not necessary to use correction factors when calculating the concentration of the standard. Otherwise, use appropriate correction factors in calculating analyte concentrations.
- 8.14. Phenol-specific System Performance Check Compounds: A methylene chloride solution of chloromethane, 1, 1-dichloroethane, bromoform, chlorobenzene, 1,1,2,2-tetrachloroethane, N-nitroso-di-n-propylamine; hexachlorocyclopentadiene; 2,4-dinitrophenol; and 4-nitrophenol, each at a concentration equal to the highest calibration standard.

9. Procedures

9.1. Standard preparation

Any one of the following four methods may be used to prepare standards under this SOP. All four are adapted from USEPA Method TO-15.

9.1.1. Dynamic dilution technique

9.1.1.1. Prepare standards by dynamic dilution of the gaseous contents of a cylinder(s) containing the gas calibration stock standards with humidified zero air using mass flow controllers and a calibration manifold.

9.1.1.1.1. Deliver the working standard from the manifold to a clean, evacuated canister using a pump and mass flow controller.

9.1.1.1.2. Alternatively, calibrate the analytical system by sampling directly from the manifold, if the flow rates are optimized to provide the desired amount of calibration standards.

9.1.1.2. Because the use of a canister as a reservoir prior to introduction into the concentration component of the analytical system resembles the procedure normally used to collect and analyze samples, this method is preferred.

9.1.1.3. Measure the flow rates of the dilution air and cylinder standards (using the same units for all), with a bubble meter or calibrated electronic flow measuring device, and calculate the concentrations of target compounds in the manifold using the dilution ratio and the original concentration of each compound, as follows:

$$\text{Manifold Concentration} = \frac{(\text{Original Concentration}) \times (\text{Standard Gas Flowrate})}{(\text{Air Flowrate}) + (\text{Standard Gas Flowrate})} \quad (1)$$

9.1.1.3.1. For example, a 1 mL/min flow of 10 ppmv standard diluted with 1,000 mL/min of humid air provides a nominal 10 ppbv mixture:

$$\text{Manifold Concentration} = \frac{(10 \text{ ppm}) \times (1 \text{ mL/min})}{(1000 \text{ mL/min}) + (1 \text{ mL/min})} = \frac{(10,000 \text{ ppb}) \times (1 \text{ mL/min})}{(1000 \text{ mL/min}) + (1 \text{ mL/min})} = 10 \text{ ppb} \quad (2)$$

9.1.2. Static dilution bottle technique

9.1.2.1. Prepare standards in canisters by spiking the canister with a mixture of liquid components prepared in a static dilution bottle.

9.1.2.2. Determine the volume of a clean 2-L round-bottom flask (modified with a threaded glass neck to accept flexible tubing or a Mininert septum cap) by weighing the amount of water that completely fills the flask. Assume a density for the water of 1 g/mL, so the weight of the water (in g) is the flask volume (in mL).

9.1.2.3. Flush the flask with helium by attaching tubing into the glass neck to deliver the helium. After a few minutes, remove the tubing, and immediately close the glass neck with a Mininert septum cap.

- 9.1.2.4. Place the flask in a 60°C oven, and allow it to equilibrate at that temperature for about 15 min. Inject a predetermined aliquot of liquid standard(s) into the flask, making sure to keep the flask temperature constant at 60°C.
- 9.1.2.4.1. Make sure that the volume of liquid standard injected into the flask does not result in an overpressure due to the higher partial pressure produced by the standard compared to the vapor pressure in the flask.
- 9.1.2.5. Allow the contents of the flask to equilibrate in the oven for at least 30 min.
- 9.1.2.6. To avoid condensation during withdrawal of aliquots from the flask, place the syringes that will be used to make withdrawals in the oven at the same temperature to preheat them.
- 9.1.2.7. Withdraw sample aliquots for introduction into the analytical system, or for further dilution. Avoid taking an aliquot (or aliquots) totaling greater than 1% of the flask volume (i.e. ~ 20 mL).
- 9.1.2.8. Standards prepared by this method are stable for one week. Replace the septum with each freshly prepared standard.
- 9.1.2.9. Use the following equation to calculate the concentration of each standard component in the flask:

$$\text{Concentration (mg/L)} = \frac{(V_a)(d)}{V_f} \quad (3)$$

Where:

- V_a = Volume of liquid neat standard injected into the flask (μL)
 d = Density of the liquid neat standard ($\text{mg}/\mu\text{L}$)
 V_f = Flask volume (L)

- 9.1.2.9.1. To obtain concentrations in ppbv, divide the gaseous volume of the injected compound by the cylinder volume (at STP) and then multiply by 10^9 .
- 9.1.3. Standard preparation in high-pressure cylinders
- 9.1.3.1. Obtain standard compound(s) as gas(es) or neat liquid(s). This method is suitable for standards of $\geq 98\%$ purity.
- 9.1.3.2. Flush an aluminum cylinder with high-purity nitrogen gas (Section 8.10) and evacuate it to a pressure ≤ 25 in of Hg. The cylinder should be equipped with a heated injection port and nitrogen flow to facilitate sample transfer.
- 9.1.3.3. Using a microliter or gastight syringe, measure predetermined amounts of each neat standard compound, and inject them into the cylinder.
- 9.1.3.4. Pressurize the cylinder to 1000 psig with high-purity nitrogen.
- 9.1.3.5. Allow the contents of the cylinder to equilibrate (~ 24 h) prior to withdrawing any aliquots for analysis.
- 9.1.3.6. If the neat standard is a gas, determine the cylinder concentration using the following equation (note that all volumes must be in the same units):

$$\text{Concentration (ppbv)} = \frac{\text{Volume}_{\text{standard}}}{\text{Volume}_{\text{dilution gas}}} \times 10^9 \quad (4)$$

9.1.3.7. If the neat standard is a liquid, determine the gaseous volume using the equations

$$V = \frac{nRT}{P} \quad \text{and} \quad n = \frac{(V_L)(d)}{MW} \quad (5)$$

Where:

V = Gaseous volume (L) at EPA standard temperature (25°C) and pressure (760 mm Hg)

n = Moles

R = Gas constant (0.08206 L-atm/mole °K)

T = 298°K (standard temperature).

P = 1 standard pressure, 760 mm Hg (1 atm)

V_L = Volume of liquid injected (mL)

d = Density of the neat standard (g/mL)

MW = Molecular weight of the neat standard expressed (g/mole)

9.1.3.8. Divide the gaseous volume of the injected compound by the cylinder volume (at STP), and multiply by 10⁹ to obtain the concentration in units of ppb.

9.1.4. Standard preparation by water methods

9.1.4.1. Evacuate a previously cleaned canister, and pressurize to 760 mm Hg absolute (1 atm) with zero grade air.

9.1.4.2. Remove the air gauge from the canister, and connect a sparging vessel to the canister with a short length of 1/16" stainless steel tubing.

9.1.4.2.1. Minimize dead volume to maximize analyte transfer from water to canister.

9.1.4.3. Spike a predetermined amount of the stock standard solution, and the internal standard solution, into 5 mL of water.

9.1.4.4. Transfer this water into the sparge vessel, and purge with zero grade nitrogen for 10 min at 100 mL/min, while maintaining the sparging vessel at 40°C.

9.1.4.5. Remove the sparge vessel and replace the air gauge on the canister.

9.1.4.6. Pressurize the canister with high-purity nitrogen to 1500 mm Hg absolute pressure (approximately 29 psi absolute).

9.1.4.7. Allow the canister to equilibrate overnight before use.

9.2. System settings

- 9.2.1. Use the general GC/MS operating conditions (Table 2), taken from USEPA Methods 8270C and TO-15, as guidance for setting the various GC/MS parameters:

Table 2. GC-MS operating settings and parameters.

Mass range	35-500 amu
Scan time	1 s/scan
Initial temperature	40°C, hold for 4 min
Temperature program	40-270°C at 10 °C/min
Final temperature:	Highest temperature in above gradient, hold for 5 min
Injector temperature	250-300°C**
Transfer line temperature	250-300°C**
Source temperature	According to manufacturer's specifications
Injector	Grob-type, splitless*
Injection volume	1-2 µL
Carrier gas	Helium (30 cm/s)
Ion trap only	Set axial modulation, manifold temperature, and emission current to manufacturer's recommendations

* Split injection is allowed if the sensitivity of the mass spectrometer is sufficient.

** These temperatures should be higher than highest temperature in the temperature program.

9.3. Initial tuning, initial calibration, and system performance check

9.3.1. Initial tuning and calibration

- 9.3.1.1. Use Perfluorotributylamine (PFTBA) as the tuning compound along with a built-in calibrant delivery system to perform Autotune (standard spectra tune) as initial tuning or after any maintenance.
- 9.3.1.2. Quicktune should be performed daily at the beginning of each batch of samples (i.e. once per 24-h time period of operation).
- 9.3.1.3. Use the following as tuning acceptance criteria for the PFTBA standard:
- 9.3.1.3.1. The ratio of the ion at m/z 69 to that at m/z 70 must be 1 percent (± 0.50 %).
- 9.3.1.3.2. The ratio of the ion at m/z 219 to that at m/z 220 must be 5 percent (± 1.25 %).
- 9.3.1.3.3. The ratio of the ion at m/z 502 to that at m/z 503 must be 10 percent (± 1.0 %).
- 9.3.1.3.4. The peak width at half-height for the ions at m/z 69, 219, and 502 must be 0.5 amu (± 0.2 amu).
- 9.3.1.3.5. The mass assignments for the ions at m/z 69, 219, and 502 must be correct to within 0.1 amu (e.g., 69.0 amu ± 0.1 amu).
- 9.3.1.4. Alternatively, use other documented tuning criteria (e.g. manufacturer's instructions), provided that performance is not adversely affected.
- 9.3.1.5. Special Autotune can be performed to meet the tuning criteria of decafluorotriphenylphosphine (DFTPP) and 4-bromofluorobenzene (BFB) required by EPA for semivolatiles and volatiles.
- 9.3.1.6. Do not begin analyses until the tuning criteria are met.

9.3.2. Injection port inertness check

- 9.3.2.1. Prepare a methylene chloride solution containing 50 ng/μL each of 4,4'-DDT, Pentachlorophenol, and benzidine to verify injection port inertness and GC column performance. Store solution at -10°C (or below) when not in use.
- 9.3.2.2. Use the DDT, pentachlorophenol, and benzidine in this standard to assess column performance and injection port inertness. Degradation of DDT to DDE and DDD should not exceed 20%. Benzidine and pentachlorophenol should be present at their normal area or height responses, and no peak tailing should be visible.
 - 9.3.2.2.1. If DDT breakdown is > 20% and/or poor chromatography is noted, the injection port may require cleaning. Repeat until the above criteria are met.
 - 9.3.2.2.2. Calculate DDT breakdown as follows (EPA Method 8081A):

$$\% \text{ breakdown of DDT} = \frac{\text{sum of degradation peak areas (DDD + DDE)}}{\text{sum of all peak areas (DDT + DDE + DDD)}} \times 100$$

Equation (6)

9.3.3. System performance check

- 9.3.3.1. Perform a system performance check daily to ensure that minimum average RFs are met before the calibration curve is used. For this SOP, the System Performance Check Compounds (SPCCs) are: chloromethane, 1, 1-dichloroethane, bromoform, chlorobezene, and 1,1,2,2-tetrachloroethane (volatiles); N-nitroso-di-n-propylamine; hexachlorocyclopentadiene; 2,4-dinitrophenol; and 4-nitrophenol (semivolatiles).
 - 9.3.3.2. These SPCCs typically have very low RFs (0.1-0.2), and tend to decrease in response as the chromatographic system (or the standard material) begins to deteriorate. They are usually the first to show poor performance. Calculate RF for each of the four SPCCs; the minimum acceptable average RF is 0.050. This check must be met before sample analysis begins.
 - 9.3.3.3. If the minimum response factors are not met, evaluate the system and take corrective action before sample analysis begins. Possible problems include:
 - 9.3.3.3.1. Standard mixture degradation
 - 9.3.3.3.2. Injection port inlet contamination
 - 9.3.3.3.3. Contamination at the front end of the analytical column
 - 9.3.3.3.4. Reactive (i.e. non-inert) sites in the column and/or system
- ### 9.4. Sample analysis
- 9.4.1. Dilution
 - 9.4.1.1. Samples collected according to SOP V2 will have a final vacuum of 7 in. of Hg, based on a collected volume of 4.55 L.
 - 9.4.1.2. Pressurize the canister to 1 atm (29.92 in. of Hg) above ambient, using ultrahigh-purity N₂ gas (Section 8.10).
 - 9.4.1.3. This introduces a dilution factor of 2.61 (RESTEK Canister Guide 2002):

$$(29.92 + 29.92)/(29.92 - 7) = 2.61$$

- 9.4.1.4. Adjust calculations accordingly if the final (i.e. post-sampling) vacuum of a canister is other than 7 in. of Hg.
- 9.4.2. Connect the sample canister to the inlet of the GC/MS system (through the six-port chromatographic valve).
- 9.4.2.1. Place a mass flow controller on the canister, open the canister valve, and vent the canister flow past a tee inlet to the analytical system at a flow of 75 mL/min. Pull approximately 35 mL/min through the Nafion® dryer to the six-port valve.
- 9.4.2.1.1. Flow rate is not as important as acquiring a sufficient, accurate sample volume.
- 9.4.3. Preconcentration
- 9.4.3.1. Cool the Cryogenic/Adsorbent Concentrator (Section 8.7) to its set point (-100 to 150°C). Once it has reached this point, turn the six-port chromatographic valve to the “fill” position to initiate sample collection.
- 9.4.3.2. Collect sample for the appropriate amount of time necessary to concentrate the desired volume.
- 9.4.3.3. After the sample is fully loaded onto the preconcentrator trap, dry purge the concentrator with He.
- 9.4.3.4. Cycle the six-port GC valve to the “inject” position and heat the trap to 250°C (with a ramp time of approximately 1 min). The trapped analytes thermally desorb into the cryofocusing trap of the CIS, which is initially set to -30 to -50°C.
- 9.4.4. Sorption to and isolation of cryofocusing trap
- 9.4.4.1. Allow three (3) min of gas flow through the heated Cryogenic/Adsorbent Concentrator and onto the cooled cryofocusing trap.
- 9.4.4.2. Close the valve between the Cryogenic/Adsorbent Concentrator and the cryofocusing trap, and purge the Concentrator with He.
- 9.4.5. Secondary desorption
- 9.4.5.1. Rapidly (e.g. a ramp of 40°C/sec) heat the focusing trap to 250 to 300°C, and apply a flow of at least 3 mL/min of pure helium carrier gas.
- 9.4.5.2. Desorb analytes from the focusing trap in “backflush” mode, with the gas flow through the trap in the opposite direction from that used during the focusing step.
- 9.4.5.3. The GC run is initiated based on a programmed time delay after the start of thermal desorption.
- 9.4.6. GC/MS analysis
- 9.4.6.1. Appropriate operating conditions for the GC columns listed in Section 8.2 when used under this specific SOP are in Table 3:

Table 3. Operating conditions for the GC column.

Initial temperature	40°C, hold for 3 min
Temperature program	40-220 °C at 10°C/min
Final temperature:	220°C, hold for 3 min
Carrier gas	Helium (20 mL/min)

- 9.4.6.2. Operate the GC/MS system to scan the atomic mass range from 35 - 300 atomic mass units (amu).
- 9.4.6.3. Acquire at least ten scans per chromatographic peak.
- 9.4.6.4. Selected ion monitoring (SIM) is acceptable for applications requiring detection limits below the normal range of electron impact mass spectrometry. However, SIM may provide a less definitive compound identification, unless multiple ions are monitored for each analyte.
- 9.4.7. Qualitative analysis (peak identification)
 - 9.4.7.1. Identify compounds by (a) comparing retention time and (b) comparing the sample mass spectrum (after any necessary background correction) with a reference mass spectrum (Method 8270C). Conditions that must be met for identification of compounds are as follows:
 - 9.4.7.1.1. The relative retention time (RRT) of the analyte in a sample must be within ± 0.06 RRT units of the RRT of the corresponding analyte in the standard.
 - 9.4.7.1.2. The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. For the purposes of this SOP, peak selection is done by a data system target compound search routine based on the presence of a target peak, containing ions specific for the target compound, at a compound-specific retention time.
 - 9.4.7.1.2.1. The characteristic ions from the reference mass spectrum are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum.
 - 9.4.7.1.3. The relative intensities of the characteristic ions in the spectrum of the sample component must agree within 30% of the relative intensities of the same ions in the reference spectrum.
 - 9.4.7.1.3.1. For example: If an ion has an abundance of 50% in the reference spectrum, the abundance of the same ion in a sample spectrum can range between 20% and 80%.
 - 9.4.7.2. Identify structural isomers with very similar mass spectra as individual isomers only if they have sufficiently different GC retention times.
 - 9.4.7.2.1. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights.
 - 9.4.7.2.2. If this criterion is not met, identify structural isomers as isomeric pairs.
 - 9.4.7.3. Use a library search to tentatively identify sample components not matching any calibration standard (e.g. unknown or unidentified components). Guidelines for tentative identification are:
 - 9.4.7.3.1. The molecular ion(s) and all major ions in the reference spectrum (ions $> 10\%$ of the most abundant ion) should also be in the sample spectrum.
 - 9.4.7.3.2. The relative intensities of the major ions should agree within $\pm 20\%$. As an example, an ion with an abundance of 50% in the spectrum of the standard must have an abundance in the sample of between 30 and 70%.
 - 9.4.7.3.3. Ions present in the sample spectrum but not in the reference spectrum should be reviewed to determine if they may be due to background contamination or the presence of coeluting compounds.

- 9.4.7.3.4. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.
- 9.4.8. Quantitative analysis
- 9.4.8.1. Once a compound has been identified, base its quantitation upon the integrated abundance of the primary characteristic ion from the EICP.
- 9.4.8.2. Estimate the concentration of analytes that can be identified, but that are not included in the calibration standards, using the formula given in Section 11.4.8.
- 9.4.8.2.1. Take the area A_s from the total ion chromatograms, and assume the RF for the compound to be 1.
- 9.4.8.2.2. Report all concentrations derived in this way as estimates, and note which internal standard was used in the calculation for each analyte.
- 9.4.8.3. Quantitate structural isomers that produce very similar mass spectra as individual isomers if they have sufficiently different GC retention times.
- 9.4.8.3.1. Sufficient GC resolution is defined as the height of the valley between two isomer peaks being less than 25% of the sum of the two peak heights.
- 9.4.8.4. Quantitate structural isomers that do not meet this criterion for separation together as isomeric pairs.

10. Data and Records Management

- 10.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
- 10.2. Manage all data according to SOP B5.
- 10.3. Document all data and information on laboratory data sheets, and within logbooks with permanent ink, or in electronic notes files. Initial and date all corrections.

11. Quality Control and Quality Assurance

- 11.1. Carrier gas
- 11.1.1. Use inert, 99.999% or higher purity helium as the carrier gas.
- 11.1.2. Use only stainless steel or copper carrier gas lines.
- 11.1.3. Install oxygen and organic filters on the carrier gas lines supplying the analytical system. Replace these filters regularly, according to the manufacturer's instructions.
- 11.2. Reagent purity
- 11.2.1. Use only reagent-grade chemicals in all tests.
- 11.2.2. Unless otherwise indicated, ensure that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.
- 11.2.3. If any other grade of reagent is to be used, ascertain first that it is sufficiently pure that it will not decrease the accuracy of the determination.
- 11.2.4. Use only pesticide-quality (or equivalent) solvents.

11.3. Storage of working standards

- 11.3.1. Working standards prepared in canisters may be stored for 30 d in an atmosphere free of potential contaminants. Do not exceed this storage time.
- 11.3.2. Keep a storage logbook to document the date that any canister working samples are prepared, so their storage time can be tracked.

11.4. Full calibration

- 11.4.1. Do a full calibration during initial instrument setup, whenever the analytical method parameters are substantially changed, or whenever significant maintenance is performed on the system. Significant maintenance includes:
 - 11.4.1.1. Ion source cleaning or repair
 - 11.4.1.2. Injection port cleaning (e.g. change septa, o-ring, liner, and/or gold-plated seal)
 - 11.4.1.3. Column replacement
- 11.4.2. Do a full calibration whenever the system fails the daily single-point calibration check (Section 11.5) twice in one day.
- 11.4.3. Set up the dynamic calibration system (Fig. 1).
- 11.4.4. Analyze a sample of humidified zero air.
- 11.4.5. After the zero air check, begin the multipoint calibration. For each desired concentration, meter the contents of the working standard cylinder (2 mL/min) into the manifold's heated mixing chamber, where they are diluted and mixed with humidified zero air. For example, to calibrate the system at a nominal 10 ppbv, zero air flow rate would be 2 L/min. Calibrate the system at five concentrations, spanning the monitoring range. The lowest point on the calibration curve must be below the lowest concentration that meets the data quality objectives of the project, and close to the detection limit for the analyte. For example, if the range of interest is 2 to 20 ppbv, standards of 1, 2, 5, 10 and 25 ppbv are appropriate.
 - 11.4.5.1. One point of the full calibration curve must correspond to the concentration in the daily single-point calibration check standard (Section 11.5).
 - 11.4.5.2. Run the lowest concentration on the calibration curve first, and the highest last.
 - 11.4.5.3. Allow each working standard/zero air mixture to flow and equilibrate for 30 min before taking a sample for GC/MS analysis.
- 11.4.6. Run a second zero air sample, to re-check the system cleanliness and check the potential for sample carryover.
- 11.4.7. Using the primary ions for each compound (unless interferences are present, in which case a secondary ion is used), quantitate all analytes in the calibration curve.
- 11.4.8. Calculate relative response factors (RRFs) for each target compound in each standard, by dividing detector response by the nominal concentration of the analyte:

$$\text{RRF} = \frac{A_s}{C_s} \quad (7)$$

Where:

A_s = Peak area (or height) of the analyte or surrogate

C_s = Concentration of the analyte or surrogate (ppbv)

- 11.4.8.1. Calculate mean RRF (using all five standards), standard deviation and %RSD.

- 11.4.9. Calculate mean RT (using each of the five concentrations in the standard curve), as well as the standard deviation and %RSD.
- 11.4.10. Tabulate the area response (Y) of the primary ion for each compound and the corresponding concentration for each compound and internal standard.
- 11.4.11. Calculate the mean area response for each internal standard compound over the entire calibration range.
- 11.4.12. Technical acceptance criteria for the initial calibration are as follows:
- 11.4.12.1. The calculated %RSD for the RRF for each compound in the calibration table must be less than 30%. There may be at most two exceptions with %RSDs up to 40%. No compounds may have an RRF %RSD greater than 40%.
- 11.4.12.2. The RTs for each target compound at each individual calibration level must be within 6% of the mean RT for the compound.
- 11.4.12.3. The RTs for each target compound at each individual calibration level must be within 6% of the mean RT for the compound.
- 11.4.12.4. For each internal standard component, the retention time at each concentration must be within 20 s of the mean retention time over the initial calibration range.
- 11.4.13. Initial calibration acceptance criteria must be met before analyzing field samples.
- 11.4.13.1. If the initial calibration technical acceptance criteria are not met, inspect the system for problems. It may be necessary to clean the ion source, change the column, or take other corrective actions.
- 11.4.14. Do not report quantitative results for target compound(s) not included in the calibration standard(s), unless they are noted as estimates (Section 9.4.8.2).
- 11.5. Daily (single-point) calibration checks
- 11.5.1. Perform a check of the calibration curve once every 24 h – after the GC/MS system has met the tuning criteria for PFTBA, but before any actual samples are run.
- 11.5.2. Analyze the mid-level concentration from the calibration curve as the standard for the calibration check.
- 11.5.2.1. Introduce this standard through the dynamic calibration system.
- 11.5.2.2. Alternatively, the standard can be added from a certified canister that has been filled (from the dynamic calibration system) with the diluted working standard.
- 11.5.2.2.1. If this option is chosen, allow the gas flow to properly equilibrate (Section 11.4.5.3) before filling the canister.
- 11.5.2.2.2. Do not use calibration check standards that are > 30 d old (Section 6.1.7.1).
- 11.5.3. Perform the following calculations:
- 11.5.3.1. Calculate a relative response factor (RRF) for each analyte (Section 11.4.8).
- 11.5.3.2. Calculate the percent difference in the RRF of the daily RRF compared to the mean RRF in the most recent full calibration. Calculate the %D for each target compound using the following equation:

$$\%D = \frac{(RRF_c - \overline{RRF_i})}{\overline{RRF_i}} \times 100 \quad (8)$$

Where:

RRF_c = RRF of the target compound in the daily calibration check run

$\overline{RRF_i}$ = Mean RRF of the target compound in the most recent full calibration

- 11.5.4. Acceptance criteria for daily calibration checks are as follows:
 - 11.5.4.1. The %D for each target compound in a daily calibration sequence must be within $\pm 30\%$ in order to proceed with the analysis of samples and blanks.
 - 11.5.4.2. Maintain a control chart showing %D values for each daily calibration check. Values for %D that steadily increase over time, although still within the acceptable range, may indicate that maintenance will soon be needed.
- 11.5.5. If the daily calibration technical acceptance criteria are not met, it may be necessary to clean the ion source, change the column, or take other corrective action(s).
 - 11.5.5.1. Rerun the daily calibration check standard. If the %D criterion still cannot be met, more thorough corrective action may be needed.
 - 11.5.5.1.1. In this case, rerun the full calibration (Section 11.4).
- 11.6. Method TO-15 also advises analyzing one calibration check standard roughly every tenth sample during an extended analytical sequence, to check system performance.
 - 11.6.1. For runs containing more than 15 samples, a calibration check standard may be run near the middle of the batch, and evaluated as per Section 11.5.4. For runs longer than approximately 30 samples, consider adding two calibration check standards.
- 11.7. Check each analytical run for saturation. The level at which a compound saturates the detection system is a function of system sensitivity and the mass spectral characteristics of that compound. Denote any analyte(s) in a particular sample that are found to be saturated as “above the quantitation limit” (with the limit identified) in that sample.
- 11.8. Use secondary ion quantitation only when there are sample matrix interferences with the primary ion. If secondary ion quantitation is performed, document the reasons.
- 11.9. Blanks
 - 11.9.1. **Laboratory blanks** are identically prepared (cleaned and certified) canisters, with similar histories and conditioned at the same time as those used for sampling, pressurized to 1 atm with N₂ gas.
 - 11.9.1.1. Include at least two laboratory blanks per sampling event.
 - 11.9.1.2. Store laboratory blanks in clean controlled conditions prior to use.
 - 11.9.1.3. Analyze laboratory blanks at the same time as the samples (after the calibration check) - one at the beginning and one at the end of the sequence of runs.
 - 11.9.1.4. Add any internal standard to laboratory blanks in the same mass and volume added to samples.
 - 11.9.1.5. Acceptance criteria for laboratory blanks are as follows:
 - 11.9.1.5.1. The blank should not contain any target analyte at a concentration greater than its quantitation level (three times the MDL, Section 11.10).
 - 11.9.1.5.2. The blank should also not contain any additional (non-target) compounds with retention times and mass-spectral features that would interfere with identification and measurement of a method analyte.
 - 11.9.1.6. If the blanks do not meet the technical acceptance criteria, investigate the source of the contamination and take (and document) appropriate corrective measures before proceeding with further sample analysis.
 - 11.9.1.7. Whenever a particularly concentrated sample is encountered (i.e., one outside the calibration range), analyze a blank canister immediately after the sample is completed, as a check against carryover effects.

- 11.9.2. **Field blanks** are the same as laboratory blanks; however, they are transported to and from the monitoring site. They do not actually receive air.
 - 11.9.2.1. Take one field blank canister for every ten canisters included in a sampling event, with the exception that no less than two field blanks may be included, however small the study.
 - 11.9.2.2. Distribute field blanks evenly throughout the run when the canisters are analyzed.
- 11.10. Method detection limits
 - 11.10.1. Determine the method detection limit for each compound on a particular GC/MS system by making seven replicate measurements of the compound of interest at a concentration near (within a factor of five) the expected detection limit. Calculate the standard deviation for the seven replicate concentrations, and multiply it by 3.14 (the Student's *t* value for 99 percent confidence for seven values).
- 11.11. System precision checks
 - 11.11.1. Test analytical system precision using six calibration check standard canisters.
 - 11.11.2. Conduct system precision checks once every tenth batch of samples or once every three months, whichever happens first.
 - 11.11.3. Conduct system precision checks whenever the analytical method is modified.
 - 11.11.4. Include the report produced from the most recent precision test with the report generated for each batch of samples.
- 11.12. Initial demonstration of analyst proficiency
 - 11.12.1. Whenever new analysts are trained to conduct the analyses described in this SOP, they will be required to successfully complete all of the calibrations, performance checks, and verifications outlined in this SOP prior to generating actual data.
- 11.13. Modifications or changes to the system
 - 11.13.1. If any changes are made to the system (e.g., the column is changed, a septum is changed), the following guidance (from Section 8.2 of USEPA Method 8000B) dictates whether or not recalibration of the system is necessary.
 - 11.13.1.1. The following procedures will automatically require recalibration of the system:
 - 11.13.1.1.1. Changing, replacing, or reversing the column
 - 11.13.1.1.2. Cleaning the MS source
 - 11.13.1.1.3. Changing the electron multiplier, ion source chamber, or injector port
 - 11.13.1.2. The following procedures will probably not require recalibration of the system, but must still be followed by a calibration check standard analysis:
 - 11.13.1.2.1. Changing septa or split seals
 - 11.13.1.2.2. Changing compressed gas cylinders and/or PTFE transfer lines
 - 11.13.1.2.3. Changing syringes
 - 11.13.1.2.4. Changing moisture, hydrocarbon, or oxygen traps
 - 11.13.1.2.5. Changing column fittings, inlets, or filaments
 - 11.13.1.2.6. Cleaning the inlet port
 - 11.13.1.2.7. Breaking off or changing a guard column

12. References

- 12.1. USEPA TO-14A. 1999. Determination of Volatile Organic Compounds (VOCs) in Ambient Air Using Specially Prepared Canisters with Subsequent Analysis by Gas Chromatography. In: Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, 2nd Ed. (EPA/625/R-96/010b). Center for Environmental Research Information, Office of Research and Development, Cincinnati, OH.
- 12.2. USEPA Method TO-15. 1999. Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS). In: Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition (EPA/625/R-96/010b). Center for Environmental Research Information, Office of Research and Development, Cincinnati, OH.
- 12.3. USEPA Method TO-17. 1999. Determination of Volatile Organic Compounds in Ambient Air Using Active Sampling onto Sorbent Tubes. In: Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition (EPA/625/R-96/010b). Center for Environmental Research Information, Office of Research and Development, Cincinnati, OH.
- 12.4. USEPA Method 8260B. 1996. Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS). SW-846 - Test Methods for Evaluating Solid Waste: Physical/Chemical Methods. Office of Solid Waste. Washington, DC. [http://www.epa.gov/epaoswer/hazwaste/test/8_series.htm]
- 12.5. USEPA Method 8270C. 1996. Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS). SW-846 - Test Methods for Evaluating Solid Waste: Physical/Chemical Methods. Office of Solid Waste. Washington, DC.
- 12.6. California EPA SOP 059. 2003. Standard Operating Procedure for Determination of Oxygenated Hydrocarbons (OHCs) in Ambient Air by Capillary Column Gas Chromatography/Mass Spectrometry. Air Resources Board, Northern Laboratory Branch, Monitoring and Laboratory Division.
- 12.7. Colorado Department of Public Health and the Environment. 2000. Guidance for Analysis of Indoor Air Samples. Hazardous Materials and Waste Management Division, Denver, CO. Online at <http://www.cdphe.state.co.us/hm/airsmpl.pdf>. Accessed 3/30/2006.
- 12.8. GERSTEL. 2000. Operation Manual - TDS G Online Thermodesorption System in conjunction with a GC HP6890 and a GERSTEL CIS 4 Cold Injection System. GERSTEL, Inc., Mulheim an der Ruhr, Germany
- 12.9. Cai, L., J.A. Koziel, Y.-C. Lo, and S.J. Hoff. 2006. Characterization of volatile organic compounds and odorants associated with swine barn particulate matter using solid-phase microextraction and gas chromatography-mass spectrometry-olfactometry. *Journal of Chromatography A* 1102:60-72.
- 12.10. McGinn, S.M., H.H. Janzen and T. Coates. 2003. Atmospheric ammonia, volatile fatty acids, and other odorants near beef feedlots. *Journal of Environmental Quality* 32:1173-1182.

- 12.11. Wright, D.W., D.K. Eaton, L.T. Nielsen, F.W. Kuhrt, J.A. Koziel, J.P. Spinhirne, and D.B. Parker. Multidimensional gas chromatography-olfactometry for the identification and prioritization of malodors from confined animal feeding operations. *Journal of Agricultural and Food Chemistry* 53:8663-8672.
- 12.12. Alltech Associates. 1997. Phenols by Heliflex® AT-1. Online at http://www.alltechweb.com/chromaccess/chromview_prn.asp?refno=1256. Accessed 2/22/2006.
- 12.13. Alltech Associates. 1997. Volatile acids by Heliflex® AT-1000. Online at http://www.alltechweb.com/chromaccess/chromview_prn.asp?refno=1243. Accessed 2/22/2006.
- 12.14. Agilent Technologies. 2000. Agilent J&W GC Columns: DB-XLB Columns. Online at <http://www.chem.agilent.com/Scripts/PDS.asp?lPage=1248>. Accessed 3/30/2006.
- 12.15. Sigma-Aldrich. 1998. Analyzing Fatty Acids by Capillary Gas Chromatography (Supelco Bulletin 855B). Online at <http://www.sigmaaldrich.com/Graphics/Supelco/objects/4600/4509.pdf>. Accessed 3/02/2006.
- 12.16. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.
- 12.17. SOP V2. 2006. VOC Sampling Using Canisters. Standard Operating Procedure V2. Purdue Ag Air Quality Lab.
- 12.18. SOP V7. 2006. VOC Cleaning, Certification and Pre-Sampling Preparation of Sampling Canisters. Standard Operating Procedure V7. Purdue Ag Air Quality Lab.

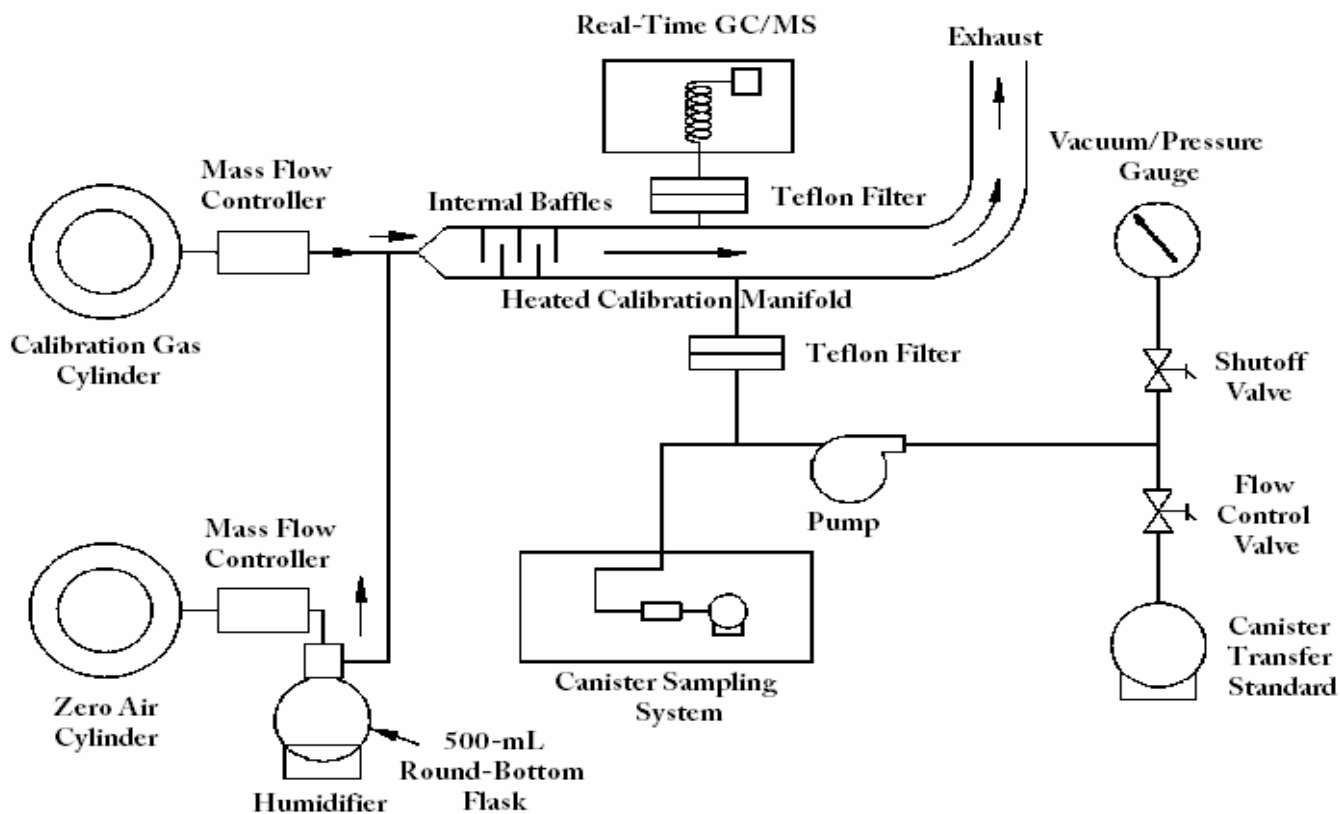


Figure 1. Schematic representation of a dynamic calibration system, which can be used for testing cleanliness of a sampling train (See SOP V7), calibrating the GC/MS (This SOP), or preparing canister transfer standards through dynamic dilution (Section 9.1.1). Adopted from USEPA Compendium Method 14A.

**CLEANING, CERTIFICATION AND PRE-SAMPLING
PREPARATION OF SAMPLING CANISTERS
Standard Operating Procedure (SOP) V7**

**CLEANING, CERTIFICATION AND PRE-SAMPLING
PREPARATION OF SAMPLING CANISTERS
Standard Operating Procedure (SOP) V7**

Prepared by

Hua Xu and Bill W. Bogan

Reviewed by

Albert J. Heber

Effective Date: November 13, 2007

PURDUE AGRICULTURAL AIR QUALITY LABORATORY (PAAQL)

Agricultural and Biological Engineering, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability.....	3
2. Summary of Method.....	3
3. Definitions.....	4
4. Health and Safety.....	4
5. Cautions.....	4
6. Interferences.....	5
7. Personnel Qualifications	6
8. Equipment and Supplies	6
9. Procedure.....	8
10. Data and Records Management	14
11. Quality Control and Quality Assurance.....	15
12. References.....	16

1. Scope and Applicability

- 1.1. A common method for sampling of volatile organic compounds (VOCs) utilizes canisters, which are evacuated to sub-atmospheric pressure in the laboratory, and then opened in the field to collect an air sample containing air pollutants of interest. Sample collection at barns and livestock buildings using this method is described in SOP V2. Analysis of the samples, including a list of target analytes which can be quantitated using these methods, is provided in SOP V6.
- 1.2. The canister must be very clean prior to sampling in the field. This SOP describes the protocols for cleaning canisters, for verifying their acceptability using GC/MS (certification), and record-keeping practices for tracking canister status.
- 1.3. This protocol is adapted from USEPA Methods TO-14A and TO-15, and ASTM Standard D5466-01.

2. Summary of Method

Fig. 1 presents a flowchart of the preparation of canisters and sampling trains for use in air sampling. Procedures for the sampling itself are described in SOP V2.

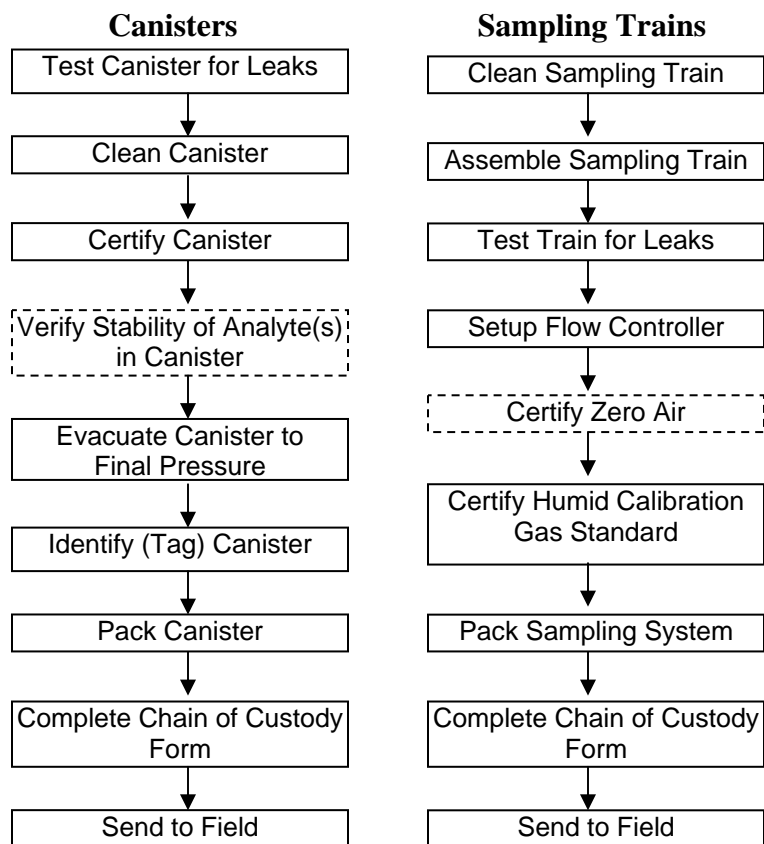


Figure 1. VOC sampling flowchart, adopted from USEPA Method TO-15. Dashed procedures are optional, or are done periodically.

3. Definitions

- | | |
|--------------------------|--|
| 3.1. ASTM | American Society for Testing and Materials |
| 3.2. Certification | The process of demonstrating with humid zero air and humid calibration gases that the sampling systems components and the canister will not change the concentrations of sampled and stored gases |
| 3.3. COC | Chain of custody |
| 3.4. GC/MS | Gas chromatography/mass spectrometry |
| 3.5. HPLC | High-performance liquid chromatography |
| 3.6. Internal standard | A pure analyte which is added to a sample (or standard) in a known amount, and is used to measure the relative responses of target compounds in the solution. The internal standard(s) must not already be present in the sample. |
| 3.7. ppbv | Parts per billion (volume basis) |
| 3.8. "Practice" Canister | A canister used to calibrate the mass flow controller. |
| 3.9. psi | Pounds per square inch |
| 3.10. QAPP | Quality Assurance Project Plan |
| 3.11. sccm | Standard cubic centimeters per minute |
| 3.12. Target analyte | A compound designated by the QAPP or Statement of Work for analysis (qualitative or quantitative) |
| 3.13. Tuning standard | A standard which is used to (a) verify that the MS system is operating properly and (b) guard against reactive sites in the system (especially the injection ports, lines, and the first segments of the column) which might cause chemical breakdown or degradation of analytes in the sample |
| 3.14. VOC | Volatile organic compound(s) |

4. Health and Safety

- 4.1. Release or evacuate air from a canister to a fume hood or to outside.
- 4.2. Because some suppliers send canisters in a pressurized state, always wear safety glasses when handling canisters.
- 4.3. To avoid burns, exercise care when using heat bands or jackets in the canister-cleaning process.

5. Cautions

- 5.1. New or reconditioned canisters should be filled with humidified Ultra High Purity (UHP) air or nitrogen and then analyzed after 24 h to evaluate cleanliness. Although the 24-h period is not a method requirement, new and reconditioned canisters have a higher potential for contamination due to the manufacturing processes, and it is therefore prudent to allow the humidified UHP air or nitrogen to remain in the canister for a longer period to ensure that contaminants are desorbed from active sites.

- 5.2. Avoid impurities from:
 - 5.2.1. Calibration gas(es)
 - 5.2.2. Internal or tuning standard dilution and/or carrier gas(es)
 - 5.2.3. Organic compounds outgassing from system components ahead of the canister
 - 5.2.4. Solvent vapors in the laboratory
- 5.3. Never use non-chromatographic grade stainless steel tubing, non-PTFE thread sealants, or flow controllers with Buna-N rubber components.
- 5.4. Dust particles can obstruct the canister orifice, compromise the leak integrity of the valve, and possibly damage the valve.
- 5.5. Clean sampling train parts with solvents in a place that guarantees no cross-contamination of the cleaned parts (i.e., inside a lab hood or in a room other than the room where the sampling train parts are assembled).
- 5.6. Once the flow rate of a sampling train has been set and certified, the train must be handled carefully, to avoid disturbing that flow rate.

6. Interferences

- 6.1. The following loss mechanisms for analytes collected into sampling canisters are generally considered (USEPA Method TO-15) to be short-term, and could affect the stability of analytes over the time periods relevant to this SOP (i.e. the two weeks required to assess analyte stability in Section 11.8). In general, however, most VOCs can be recovered from canisters near their original concentrations up to 30 d.
 - 6.1.1. Reactive sites on the internal surface(s) of the canister and/or sampling train can cause loss of analytes.
 - 6.1.2. Losses of analytes due to physical adsorption can occur. These slow as equilibrium is reached between the condensed (adsorbed) and gas phases of an analyte. These are generally considered to occur over minutes or hours, and may be more significant with low-humidity samples.
 - 6.1.3. Conversely, if there is any water condensed in the canister, analytes can be lost as they dissolve in this water. This can be particularly significant if high-humidity samples are pressurized, leading to condensation.
 - 6.1.3.1. Loss mechanisms based on aqueous hydrolysis also exist.
 - 6.1.4. Chemical reactions between VOCs and other, more stable substances inside the canister are generally assumed to cause the gradual decrease of concentration over time (i.e., long-term losses over days to weeks).
- 6.2. Biodegradation of analytes can also occur over time.
- 6.3. Impurities in the canister pressurizing gas, dilution gas (if used in the introduction of analytes during stability checks) and carrier gas, organic compounds out-gassing from the cleaning system components after cryogen traps, and solvent vapors in the laboratory can all result in contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running humidified zero air blanks, which must be shown (Section 11.1) to have less than 0.2 ppbv of any target analyte.

7. Personnel Qualifications

- 7.1. Each person working with sampling canisters must read and understand this SOP.

8. Equipment and Supplies

8.1. Equipment

- 8.1.1. Stainless steel canister(s): Leak-free stainless steel pressure vessels of desired volume with valve and specially prepared (passivated) interior surface. For the purposes of this SOP, SilcoCan[®] canisters (or equivalent) will be used.
- 8.1.2. Canister mass flow controller (Veriflo[™] Flow Controller Model SC423XL, RESTEK Corporation, Bellefonte, PA, or equivalent). Capable of maintaining a constant flow rate ($\pm 10\%$) over a sampling period of up to 24 h and under conditions of changing temperature (20°C – 40°C) and humidity. Veriflo[™] controllers contain built-in critical orifices, and the exact part number depends upon the desired flow rate of sample into the canister (SOP V2).
- 8.1.2.1. The flowrate can be calculated (SOP V2) by

$$F = \frac{P \times V}{T \times 60 \text{ (min-atm/hour)}}$$

Where:

F = flowrate, mL/min

P = final canister absolute pressure

V = volume of canister, mL

T = sample period, h

- 8.1.3. Vacuum gauge (RESTEK Catalog #24269, or equivalent)
- 8.1.4. Leak-free stainless steel valve for vacuum/pressure gauge
- 8.1.5. In-line filter cartridge (2- μm pore size, sintered stainless steel in-line filter, Nupro Co., OH, Model SS-2F-K4-2, or equivalent)
- 8.1.6. Sample inlet
- 8.1.6.1. 1/4" nut to serve as a "rain cap" at the end of the sampling inlet
- 8.1.7. Passivated stainless steel sampling tube, typically 1/4" ID,
- 8.1.8. A canister-cleaning system (Fig. 2) consisting of the following components:
- 8.1.8.1. UHP gas cylinders (1), with appropriate regulators (SOP G2). Use humidified UHP nitrogen for SilcoCans[®], and humidified zero air for SUMMA[®] canisters; In this SOP, since SilcoCan[®] will be used, UHP nitrogen is appropriate. The QAPP for a particular project will specify a source/distributor if a specific product is to be used.
- 8.1.8.1.1. Note: All UHP gas cylinders must be certified as clean (less than 0.2 ppbv of any target VOC) before they can be used in the canister-cleaning procedure.
- 8.1.8.2. A vacuum pump capable of evacuating canister(s) to an absolute pressure of <0.002 in Hg (50 mTorr) (DuraVane Two Stage High Vacuum Rotary Vane Pump (7.5×10^{-3} Torr), Part #DERVVR003H, LACO Technologies)
- 8.1.8.3. Stainless steel manifold with connections for six (6) canisters

- 8.1.8.4. Shut-off valves: Swagelok plug valves
 - 8.1.8.5. Stainless steel vacuum gauge: Capable of measuring vacuum in the manifold to an absolute pressure of 0.002 in Hg (50 mTorr) or less. All the wetted surfaces of the sensor must be of passivated stainless steel.
 - 8.1.8.6. Cryogenic traps (1): Stainless steel Dry Ice Trap (Part #LIT-10025, LACO Technologies, UT), to capture contaminants (e.g. oil from vacuum pump)
 - 8.1.8.7. Vacuum guage (Part #KJL-205, Lesker, PA) which can measure absolute pressure less than 50 mTorr.
 - 8.1.8.8. Humidification chamber (Part #24282, Restek, PA): Pressurizable water bubbler containing high performance liquid chromatography (HPLC)-grade deionized water and capable of withstanding pressure up to 90 psi.
 - 8.1.8.9. Insulated heat jackets, bands, or equivalent, that can heat canisters to 140°C (optional)
 - 8.1.9. If canister samples are loaded directly to the GC/MS, a dynamic calibration system (Fig. 3) consisting of the following components is required:
 - 8.1.9.1. Glass calibration manifold: 1.25-cm I.D. × 66 cm, with sampling ports and internal baffles to ensure proper mixing by flow disturbance
 - 8.1.9.2. Humidifier: 500-mL impinger flask containing HPLC-grade deionized water.
 - 8.1.9.3. Electronic mass flow controllers (2) with 5 L/min and 50 mL/min ranges.
 - 8.1.9.4. Teflon filter(s) (47-mm dia), to filter particulate matter
 - 8.1.10. Vacuum oven (optional) capable of holding a temperature of 50 or 100°C
 - 8.1.11. Certified mass flow meter to verify sampling train flow controller
 - 8.1.12. Vacuum gauge (capable of measuring manifold vacuum to an absolute pressure of 0.00197 in Hg (0.000967 psi) or less).
 - 8.1.13. A “practice” canister to check the flow controller
 - 8.1.14. 9/16” wrench
 - 8.1.15. 3-mm Hex (Allen) wrench
- 8.2. Supplies
- 8.2.1. 2-micron sintered stainless steel in-line particulate matter filter
 - 8.2.2. Methanol and hexane for cleaning sampling train parts
 - 8.2.3. HPLC-grade deionized water (MilliQ water)
 - 8.2.4. UHP nitrogen
 - 8.2.4.1. Must be certified (through GC/MS analysis) to have < 0.2 ppbv of each individual target analyte.
 - 8.2.5. GC/MS system (SOP V6)
 - 8.2.5.1. Gas chromatograph (Agilent GC6890 System, or equivalent)
 - 8.2.5.2. Column: Columns that are suitable for use under this SOP are the following:
 - 8.2.5.2.1. Rtx®-1 60 m × 0.32 mm × 1.0 µm capillary column (Restek Part #10157).
 - 8.2.5.2.2. An equivalent column (or column/guard column combination) may be used if equal performance can be achieved.
 - 8.2.5.3. Mass spectrometer (Agilent MSD5975 System, or equivalent)
 - 8.2.5.4. Capillary GC/MS Interface into the MS source from the GC
 - 8.2.5.5. A computer system that allows the continuous acquisition and storage of all mass spectra. Vendor (Agilent)’s ChemStation software allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time

or scan number (Extracted Ion Current Profile (EICP)). Vendor's software also allows integrating the abundances in any EICP between specified time or scan-number limits.

- 8.2.5.5.1. The most recent version of the EPA/NIST Mass Spectral Library will be utilized.
- 8.2.5.6. Helium cylinders - 99.999% or higher purity He as the GC carrier gas
 - 8.2.5.6.1. A scrubber ("Big Universal Trap", Part # RMSH-2, Agilent), or equivalent should be used to purify the carrier gas.
- 8.2.6. Dry ice and isopropanol
- 8.2.7. Low-level standard gas mix of target analytes (approx. 10 ppmv in N₂) for stability checks, and for certifying the sampling train(s)
 - 8.2.7.1. Prepare standards according to one of the methods outlined in Section 9.1 of SOP V6, using certified clean zero air as the diluent.
- 8.2.8. Chain-of-custody (COC) forms (original and field and lab copies) and canister identification tags

9. Procedure

- 9.1. Sampling train preparation
 - 9.1.1. Cleaning sampling system components
 - 9.1.1.1. Disassemble the sampling train to clean individual components.
 - 9.1.1.2. Rinse all nonmetallic parts with HPLC-grade deionized water, and dry them in a vacuum oven at 50°C for 12 to 24 h.
 - 9.1.1.3. Clean all stainless steel parts and fittings by placing them in a beaker of methanol in an ultrasonic bath for 15 min. Repeat, with hexane as the solvent.
 - 9.1.1.4. Rinse the parts with HPLC-grade deionized water and dry them in a vacuum oven at 100°C for 12 to 24 h.
 - 9.1.1.5. Thoroughly clean the particle filter.
 - 9.1.1.5.1. Disassemble the filter.
 - 9.1.1.5.2. Remove the larger particles from the frit by blowing particle-free nitrogen through the frit from the outlet surface toward the inlet surface.
 - 9.1.1.5.3. After larger particles are removed, sonicate or rinse filter parts in methanol.
 - 9.1.1.5.4. Bake the parts in an oven at 130°C to remove any residual organic vapors.
 - 9.1.1.6. Clean orifice and flow controller using either of the following two methods:
 - 9.1.1.6.1. Disassemble the flow controller and clean all the metal parts with methanol to remove any high boiling compounds that condensed onto the wetted areas of the controller. Heat the cleaned metal parts in an oven at 130°C to remove any residual organic vapors.
 - 9.1.1.6.2. A simpler flow controller cleaning method is to use a heating jacket or heat gun to heat the components of the sampling train, while purging the system with nitrogen.
 - 9.1.1.7. Assemble the sampling train (Section 9.1.2).
 - 9.1.1.8. Purge the entire system with humid UHP nitrogen for 24 h.
 - 9.1.2. Assemble the sampling train

- 9.1.2.1. Use a 9/16" wrench to assemble the sampling train components. Tighten the parts by hand and then apply a ¼ turn with the wrench. Do not overtighten the fitting. Verify that the sampling train parts cannot be rotated by hand. Fig. 4 shows the proper assembly of the sampling train components; additional instructions pertaining to each individual component follow.
- 9.1.2.2. Sampling inlet
 - 9.1.2.2.1. If assembling the sampling train for shipment to the field:
 - 9.1.2.2.1.1. Make sure that the entrance of the sampling inlet faces downward to avoid the entrance of raindrops.
 - 9.1.2.2.1.2. Install a nut at the entrance of the inlet to prevent rain droplets from accessing the inlet edge where they can enter the sampling train with the air sample.
 - 9.1.2.2.2. If the sampling train will be used indoors, these precautions are unnecessary.
- 9.1.2.3. Particle filter
 - 9.1.2.3.1. EPA Compendium Methods TO-14A and TO-15 recommend a sintered stainless steel in-line filter with 2-micron pores (Section 8.1.5).
 - 9.1.2.3.2. Attach the particle filter to the lower end of the sampling inlet, before the critical orifice (located in the Veriflo™ flow controller).
- 9.1.2.4. Critical orifice and flow controller
 - 9.1.2.4.1. Verify that the chosen Veriflo™ controller has a critical orifice of the correct diameter for the desired sample flow rate (as calculated in SOP V2). For example, for a 6-L canister sampling during a 24-h period, the orifice diameter is 0.0012" for a flow rate range of 2 to 4 sccm (note that the specific flow rate calculated in SOP V2 for these conditions was 3.19 sccm). This will ensure an accurate and valid sample. The critical orifice fitting should have a marking indicating the orifice size.
 - 9.1.2.4.2. Attach the flow controller to the in-line filter, and set the flow of this assembly according to Section 9.1.2.5. If the critical orifice is included with the flow controller, follow the recommendations of the manufacturer.
- 9.1.2.5. Setting the flow rate with the flow controller
 - 9.1.2.5.1. Remove the protective cap from the back of the Veriflo™ Flow Controller SC423XL body.
 - 9.1.2.5.2. Connect either an evacuated canister or a vacuum source to the outlet of the sampling train (i.e. the bottom of the Veriflo™ controller).
 - 9.1.2.5.3. Connect a calibrated (NIST-traceable) flow meter (Allicat Scientific, M-50 sccm, air), rotameter, or equivalent to the inlet of the train.
 - 9.1.2.5.4. Apply a vacuum by opening the canister and/or turning on the vacuum source.
 - 9.1.2.5.5. With a 3-mm hex (Allen) wrench, adjust the piston gap screw to achieve the desired flow rate. Wait for the flow to equilibrate between adjustments.
 - 9.1.2.5.6. Replace protective cap on the back of the Veriflo™ Flow Controller body.
 - 9.1.2.5.7. Remove the vacuum source or evacuated canister.

- 9.1.2.6. Vacuum gauge
 - 9.1.2.6.1. Install a vacuum gauge to monitor canister filling. The flow controller has a minimum pressure that needs to be considered to assure that the flow is stable. This minimum pressure is 7 in. Hg (3.44 psi) for the Veriflo™ Model SC423 XL Flow Controller.
 - 9.1.2.6.2. Verify that the gauge indicates zero upon arrival.
 - 9.1.2.6.3. If the indicator does not read zero, there are two possible causes.
 - 9.1.2.6.3.1. The gauge may need to be equilibrated. This is done by taking out the rubber plug on top of the gauge.
 - 9.1.2.6.3.2. The gauge may be damaged, in which case it must be returned to the manufacturer and replaced.
 - 9.1.2.7. The sampling train is now completely assembled, and ready to be leak tested, certified, or attached to a canister for shipment to the field, as appropriate.
- 9.1.3. Leak test of sampling train – can be done either of two ways:
 - 9.1.3.1. Sampling train pressure change
 - 9.1.3.1.1. Cap the inlet, attach the sampling train to an evacuated canister, open the valve on the canister and evacuate the sampling train.
 - 9.1.3.1.2. Close the valve and monitor any pressure change in the static sampling train. If the pressure of the static sampling train remains stable for 1-2 min, the sampling train is sufficiently leak-free, and may be used.
 - 9.1.3.1.3. If the sampling train is leak-free, set the desired sampling flow rate.
 - 9.1.3.1.4. If the sampling train does leak, check every connection again, tightening as necessary, and repeat the procedures in Sections 9.1.3.1.1 and 9.1.3.1.2.
 - 9.1.3.2. Helium leak detection
 - 9.1.3.2.1. Pass helium gas through the flow controller and use a sensitive helium leak detector to test for leaks.
- 9.1.4. Certifying sampling train cleanliness: UHP nitrogen certification
 - 9.1.4.1. Certify the train by passing a humidified, UHP nitrogen stream through the train, concentrating the exit gas on the trap, and analyzing by GC/MS (SOP V6).
 - 9.1.4.1.1. The UHP nitrogen cylinder must be certified as clean (Section 8.2.4.1).
 - 9.1.4.2. Assemble the calibration system and manifold (Fig. 3). Connect the sampler (without a canister) to the manifold, and open the UHP nitrogen cylinder to generate a humid gas stream to the calibration manifold. The flow rate should be ~2 L/min.
 - 9.1.4.3. Pass the humid zero gas stream through the calibration manifold and the sampling train (without a canister) to the GC/MS analytical system at 75 mL/min, so that 35 mL/min is pulled through the six-port valve of the GC and routed through the cryogenic trap at the GC inlet at the appropriate time. Vent the rest of the sample.
 - 9.1.4.4. Following the procedures given in SOP V6, concentrate approximately 1000 mL of sample, and route this concentrated sample to the column. Conduct analysis as described in SOP V6.
 - 9.1.4.5. For the sampling train to pass certification, the analytical system (GC/MS) must not detect any target VOC analyte at or above a concentration corresponding to 0.2 ppbv in the original air stream.

- 9.1.4.5.1. If more than 0.2 ppbv is detected for any target compound, repeat the procedures 9.1.1 to 9.1.5.
- 9.1.4.5.2. If the sampling system is certified clean with zero air, it must then be tested with standards of gas in the range of concentrations expected in the field. This is done by dynamic dilution, as discussed in the following section.
- 9.1.5. Verify cleanliness of the canister(s) and their associated sampling system(s) using one of the following two methods:
 - 9.1.5.1. Sampler system certification with sorbent tubes
 - 9.1.5.1.1. Pressurize the canister(s) to be certified with UHP nitrogen.
 - 9.1.5.1.2. Connect the sampling system(s) to the canisters
 - 9.1.5.1.3. Connect a sorbent tube containing the appropriate packing material (SOP V1) to each canister/sampling system combination.
 - 9.1.5.1.4. Open the canister valve, and allow approximately 2.0 – 2.5 L of the canister contents to pass through the sorbent tube at a rate of 50 – 100 mL/min.
 - 9.1.5.1.5. Analyze the sorbent tube (SOP V4) and determine the concentration(s) of any target compound(s) detected. If any target compound is present at 0.2 ppbv or greater, the canister(s) in question must be re-cleaned and re-tested.
 - 9.1.5.2. Sampler system certification with humid calibration gas standards from a dynamic calibration system
 - 9.1.5.2.1. Assemble the dynamic calibration system and manifold (Fig. 3).
 - 9.1.5.2.2. Verify that the calibration system is clean (less than 0.2 ppbv of any target compounds) by sampling a humidified gas stream (SOP V2), without gas calibration standards (e.g. ultra-high purity zero air or nitrogen), with a canister that has been previously certified as clean. Subject this collected sample to GC/MS analysis according to SOP V6.
 - 9.1.5.2.2.1. The assembled dynamic calibration system is certified clean if less than 0.2 ppbv of any target VOC compound is found in the collected sample.
 - 9.1.5.2.3. For generating the humidified calibration standards, attach calibration gas cylinder(s) (Section 8.2.7) to the calibration system as illustrated in Fig. 3. Open the gas cylinders (gas calibration standards and zero air) and pass the gas mixtures through 0 to 10 mL/min certified mass flow controllers to generate ppb levels of calibration standards. Relative flow rates from the two gas cylinders will depend on the concentration(s) of analyte(s) in the standard.
 - 9.1.5.2.4. After equilibrating approximately 5 min, attach sampling system (containing an evacuated canister that has been certified as clean) to the manifold (Fig. 3).
 - 9.1.5.2.5. Sample the dynamic calibration gas stream (SOP V2).
 - 9.1.5.2.6. Concurrent with the sampling system operation, real-time monitoring of the calibration gas stream is accomplished by the on-line GC/MS analytical system (Fig. 3) to provide reference concentrations of generated VOCs.
 - 9.1.5.2.7. After the sampling period (normally the same length of time used for field sampling), analyze an aliquot taken from the sampling system canister (SOP V6). Compare results to reference (real-time) GC/MS analyses to determine if the sampling system changed the concentration(s) of target VOC(s).
 - 9.1.5.2.8. A recovery of between 90% and 110% is expected for all targeted VOCs.

9.1.5.2.9. The percent of recovery is calculated:

$$\% \text{ Recovery} = \frac{Y - X}{X} \times 100$$

Where:

X = concentration of VOC analyte from calibration gas (determined by real-time GC/MS), ppbv.

Y = concentration of VOC targeted compound recovered from sampler, ppbv.

9.1.6. Sampling train packing

9.1.6.1. Carefully pack the certified sampling train in aluminum foil or in a clean container for storage, or for shipment to the field site.

9.1.6.2. Packing is critical. Careless handling could affect the preset flow rate.

9.1.7. Complete the chain of custody form.

9.1.7.1. Include the original COC and the field copy in the box with the sampling train. Keep a copy in the laboratory canister file.

9.2. Canister preparation

9.2.1. Canister cleaning

9.2.1.1. Assemble a canister cleaning system (Fig. 2), consisting of the components listed in Section 8.1.3.

9.2.1.2. Add cold trap to the vacuum pump trap

9.2.1.2.1. Add 5 lb (2.3 kg) dry ice (crushed into small cubes) to the 3.3-L cold well of cold trap.

9.2.1.2.2. Add 946 mL isopropanol (at least 90% strength) to make a dry ice slurry, which can typically last for 8 h.

9.2.1.3. Connect the desired number of canister(s) to the manifold.

9.2.1.4. Open the vent shut-off valve and the canister valve(s) to release any remaining pressure in the canister(s).

9.2.1.5. Heat canisters to 120°C-140°C. (optional)

9.2.1.5.1. If using heat bands, position them around the equator of the canister(s) according to manufacturer's instructions. Note that the valve area of the canister will not be directly heated; however, the amount of radiant heat generated at the valve by heating the canister equator to this temperature should be sufficient to decontaminate the valve (Restek Corporation 2002)

9.2.1.5.2. Heat jackets can be customized to cover the canister valve.

9.2.1.6. Start the vacuum pump, close the vent shut-off valve, and open the vacuum shut-off valve.

9.2.1.7. Once the canister(s) reaches the target temperature, evacuate the canister(s) to <0.002 in Hg (50 m Torr) and maintain them at this pressure for at least 1 h.

9.2.1.8. Close the vacuum and vacuum/pressure gauge shut-off valves and open the UHP nitrogen shut-off valve to pressurize the canister(s) with humidified nitrogen. Pressurize all canister(s) to approximately 60.8 in. Hg (30 psi). Wait for 30 min.

- 9.2.1.9. Close the UHP nitrogen shut-off valve and allow the canister(s) to vent down to atmospheric pressure through the vent shut-off valve.
- 9.2.1.10. Close the vent shut-off valve.
- 9.2.1.11. Repeat Sections 9.2.1.1 through 9.2.1.10 two additional times for a total of three (3) evacuation/pressurization cycles for each set of canisters.
 - 9.2.1.11.1. Note: It is possible that canisters used under some conditions (e.g. high levels of pollutant(s) in the air stream) may need to be subjected to a higher number of cycles. Use the canister log (Section 10.4) to help evaluate whether this is likely based on how many cycles were required to clean the canister(s) after sampling similar atmospheres in the past.
- 9.2.1.12. After the required number of pressurization/heating/evacuation cycles, pressurize the canister(s) to 60.8 in. Hg (30 psig) with humid nitrogen. Initiate a leak check (Section 9.2.2) immediately.
- 9.2.1.13. Clean cold trap
 - 9.2.1.13.1. Release pressure or vacuum.
 - 9.2.1.13.2. Warm the trap to room temperature.
 - 9.2.1.13.3. Remove center well from the chamber.
 - 9.2.1.13.4. Properly dispose of the slurry.
 - 9.2.1.13.5. Wipe outside of the cold well to achieve its original pristine sheen.
 - 9.2.1.13.6. Mild soap can be used to clean the cold well, but it should be rinsed with deionized water.
 - 9.2.1.13.7. Clean acrylic lid and o-ring, using only a damp or dry cloth.
 - 9.2.1.13.8. Apply small amount of vacuum grease to o-ring
- 9.2.2. Canister leak check
 - 9.2.2.1. After pressurizing to 60.8 in. Hg (30 psig), record the initial pressure.
 - 9.2.2.2. Close the canister valve.
 - 9.2.2.3. Check the final pressure after 24 h.
 - 9.2.2.4. To be acceptable, the final pressure must not vary by more than ± 4.08 in. Hg (± 2 psi) over the 24 h.
- 9.2.3. Canister certification
 - 9.2.3.1. Assessing canister cleanliness
 - 9.2.3.1.1. Allow a minimum of 12 h of "aging" between the final pressurization step of the cleaning process (Step 9.2.2.14) and the analysis.
 - 9.2.3.1.2. Analyze the contents of the canister(s) by a GC/MS following SOP V6.
 - 9.2.3.1.3. Do not use any canister that has not tested clean (< 0.2 ppbv each of targeted compounds). Repeat the cleaning process in Section 9.2.2 until the canister achieves the required cleanliness.
 - 9.2.3.1.3.1. Note in the canister log (Section 10.4) each time a canister requires more than the normal number of cycles to clean. Comparison with the sampling history can help to identify cases in which canisters will require more extensive cleaning in the future, and will streamline the cleaning/certification process.
 - 9.2.3.1.4. Once tested clean, re-evacuate the canister(s) to < 0.002 in Hg (50 mTorr), close the canister valve(s), and keep the canister(s) in the evacuated state until used.

- 9.2.3.1.4.1. Use an accurate vacuum gauge (Section 8.1.12) to ensure that the required vacuum is obtained. Attach the vacuum gauge to the sample inlet, and open the canister valve to double-check the reading displayed on the canister's vacuum gauge (Fig. 4).
- 9.2.3.1.5. Remove the canister from the cleaning system and cap the canister connection(s) with stainless steel fitting(s).
- 9.2.3.2. The canister is now ready for collection of an air sample.
- 9.2.4. Canister identification
 - 9.2.4.1. Attach an identification tag to each canister. This tag includes, at minimum, spaces for the following information, which must match information on the COC:
 - 9.2.4.1.1. Canister serial number
 - 9.2.4.1.2. Sample number (will be filled out in the field)
 - 9.2.4.1.3. Location (will be filled out in the field)
 - 9.2.4.1.4. Date (will be filled out in the field)
 - 9.2.4.1.5. Initial/final pressure (will be filled out in the field)
 - 9.2.5. Canister packing for shipment
 - 9.2.5.1. Pack canister carefully. Several canisters can be packed in one box.
 - 9.2.6. Canister Chain of Custody
 - 9.2.6.1. Complete the canister COC form.
 - 9.2.6.2. Include the original and field copies in the box with the canister.
 - 9.2.6.3. Keep a copy in the lab canister file.
- 9.3. Troubleshooting
 - 9.3.1. If there is no flow through the sampling train:
 - 9.3.1.1. Check that the filter is not clogged with particles. Clean (Section 9.1.1.5) if necessary.
 - 9.3.1.2. Check the orifice to verify it is not obstructed with particles. This is more critical when using very small orifices.

10. Data and Records Management

- 10.1. Maintain all lab records in a worksheet of the electronic field notes spreadsheet designated for this method. Supplement this electronic record by a bound record book designated for the method, which should contain copies of the electronic record.
- 10.2. Manage all data according to SOP B5.
- 10.3. Document all data and information on field (or laboratory) data sheets, and within site (or laboratory) logbooks with permanent ink, or in electronic field notes.
- 10.4. Maintain a permanent log for each individual canister and sampling system, which must include, at minimum, the following information:
 - 10.4.1. Dates and parameters of all cleanings (i.e. temperature applied, number of cycles required to achieve certification)
 - 10.4.2. Certification dates
 - 10.4.3. Data (i.e. chromatograms) on which each certification of cleanliness is based
 - 10.4.4. Dates and results of all leak tests
 - 10.4.5. Dates and results of all analyte stability checks

10.4.6. Dates and locations of all sampling events, as well as analytical results obtained from each sample

10.4.7. Dates and results of all dynamic calibration tests for sampling systems

11. Quality Control and Quality Assurance

- 11.1. The analytical system must be certified clean at the initiation of a project. Using a volume of UHP nitrogen equivalent to the maximum sample volume expected to be used, a GC/MS run (conducted according to SOP V6) must result in less than 0.2 ppbv of each individual target analyte.
- 11.2. Test each new UHP nitrogen cylinder to ensure that it meets this same level of purity (less than 0.2 ppbv of each individual target analyte). Otherwise, do not use the cylinder for any procedure in this SOP.
- 11.3. Calibrate and leak-check all flow controllers annually.
- 11.4. Subject all sampling trains to a cleanliness check with zero air (Section 9.1.4) before their initial use. The required level of purity is less than 0.2 ppbv of each individual target analyte
- 11.5. Subject all sampling trains to a dynamic calibration test (Section 9.1.5) before their initial use. Recoveries of all targeted VOCs must be between 90% and 110%. Otherwise, the field sampler must be removed from use, cleaned, and recertified according to the initial certification procedures (Sections 9.1.4 and 9.1.5).
- 11.6. Perform leak checks on every canister (Section 9.2.2) and every sampling train (Section 9.1.3) after every cleaning. Pressurized canisters must not change pressure by more than ± 4.08 in. Hg (± 2 psi) over a 24-h period, and sampling trains attached to evacuated canisters must display a stable pressure for a minimum of 1-2 minutes.
- 11.7. Calibrate all mass flow meters at the beginning of the project using an ISO-certified outside lab or NIST-traceable airflow calibrators, and maintain the certificates or documentation of calibration for the duration of the project.
- 11.8. Incorporate “spot-checks” into canister and sampling train certifications
 - 11.8.1. Initially (i.e. at the start of a project), check all canisters individually by GC/MS during certification, and certify all sampling trains (Sections 9.1.4 and 9.1.5).
 - 11.8.2. After the cleanup system has been proven reliable, spot-check a lower percentage of canisters and sampling systems, and certify the entire batch.
 - 11.8.2.1. Check at least 10% of any given batch of canisters, where “batch” refers to the group of canisters cleaned together in a given session.
 - 11.8.2.2. Canisters in a batch which is to be spot-checked should have contained similar contaminant levels as a result of their most recent sample collection. If some canisters in a batch are known to have contained higher levels of contaminants than others, give preference to checking some (or all) of these.
 - 11.8.2.3. Re-check each sampling system after every tenth use, using both the zero-air and dynamic calibration tests.
- 11.9. Verify analyte stability on all new canisters (after dynamic calibration), and on each canister after every tenth sampling event using that canister.

- 11.9.1. Introduce a low working concentration of a characterized test mix into the canister. The test mix must contain all or most of the target analytes, and be prepared according to one of the methods described in SOP V6 (Section 9.1).
- 11.9.2. Analyze an aliquot of the contents of the canister immediately after introducing the test mixture and after 5, 10, 20 and 30 days.
- 11.9.3. Responses (i.e. the concentrations of any single target analyte) should not decrease more than 20% over this period.
 - 11.9.3.1. Re-clean and retest any canister that fails this test.
 - 11.9.3.2. Remove from service any canister that, while certified clean, fails the test twice.
- 11.10. Measure the pressure of the canister at the following events:
 - 11.10.1. In the lab, before it is sent to the field. Pressure should be 0.002 in Hg (50 mTorr) or less. Check this value in the field (SOP V2) to ensure that no leaks occurred in transit.
 - 11.10.2. When the canister is returned to the lab after sampling, the vacuum should be the same as the value reported in the field (i.e. noted on the COC form). If it is not, invalidate the canister, as a leak occurred during transport to the lab.

12. References

- 12.1. ASTM Method D5466-01. 2006. Standard Test Method for Determination of Volatile Organic Chemicals in Atmospheres (Canister Sampling Methodology). ASTM International, West Conshohocken, PA.
- 12.2. EPA Method TO-14A. 1999. Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS). In Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition (EPA/625/R-96/010b). Center for Environmental Research Information, Office of Research and Development, USEPA, Cincinnati, OH.
- 12.3. EPA Method TO-15. 1999. Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS). In Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition (EPA/625/R-96/010b). Center for Environmental Research Information, Office of Research and Development, USEPA, Cincinnati, OH.
- 12.4. EPA Method TO-12. 1999. Method for the Determination of Non-Methane Organic Compounds (NMOC) in Ambient Air Using Cryogenic Preconcentration and Direct Flame Ionization Detection (PDFID). In Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition (EPA/625/R-96/010b). Center for Environmental Research Information, Office of Research and Development, USEPA, Cincinnati, OH.
- 12.5. RESTEK Catalog 59977B. 2002. A guide to Passive Air Sampling Using Canisters. RESTEK Corporation, Bellefonte, PA. Online at <http://www.restek.com/restek/images/external/59977A.pdf>. Accessed on 3/20/06.
- 12.6. SOP B5. 2006. Data Management for Barns. Standard Operating Procedure B5. Purdue Ag Air Quality Lab.

- 12.7. SOP G2. 2006. Compressed Gas Cylinders. Standard Operating Procedure G2. Purdue Ag Air Quality Lab.
- 12.8. SOP V2. 2006. VOC Sampling Using Canisters. Standard Operating Procedure V2. Purdue Ag Air Quality Lab.
- 12.9. SOP V6. 2006. GC/MS Analysis of Phenols and Volatile Fatty Acids (VFAs) Collected in Sampling Canisters. Standard Operating Procedure V6. Purdue Ag Air Quality Lab.

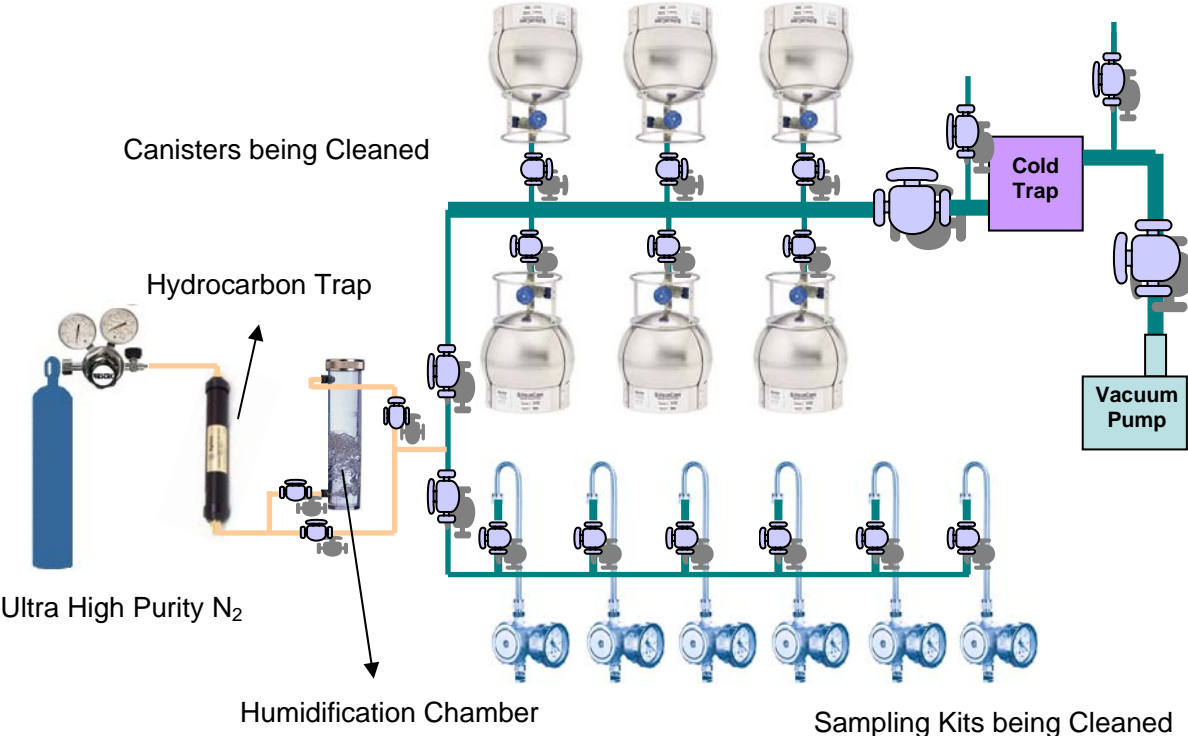


Figure 2. Schematic of canister-cleaning system.

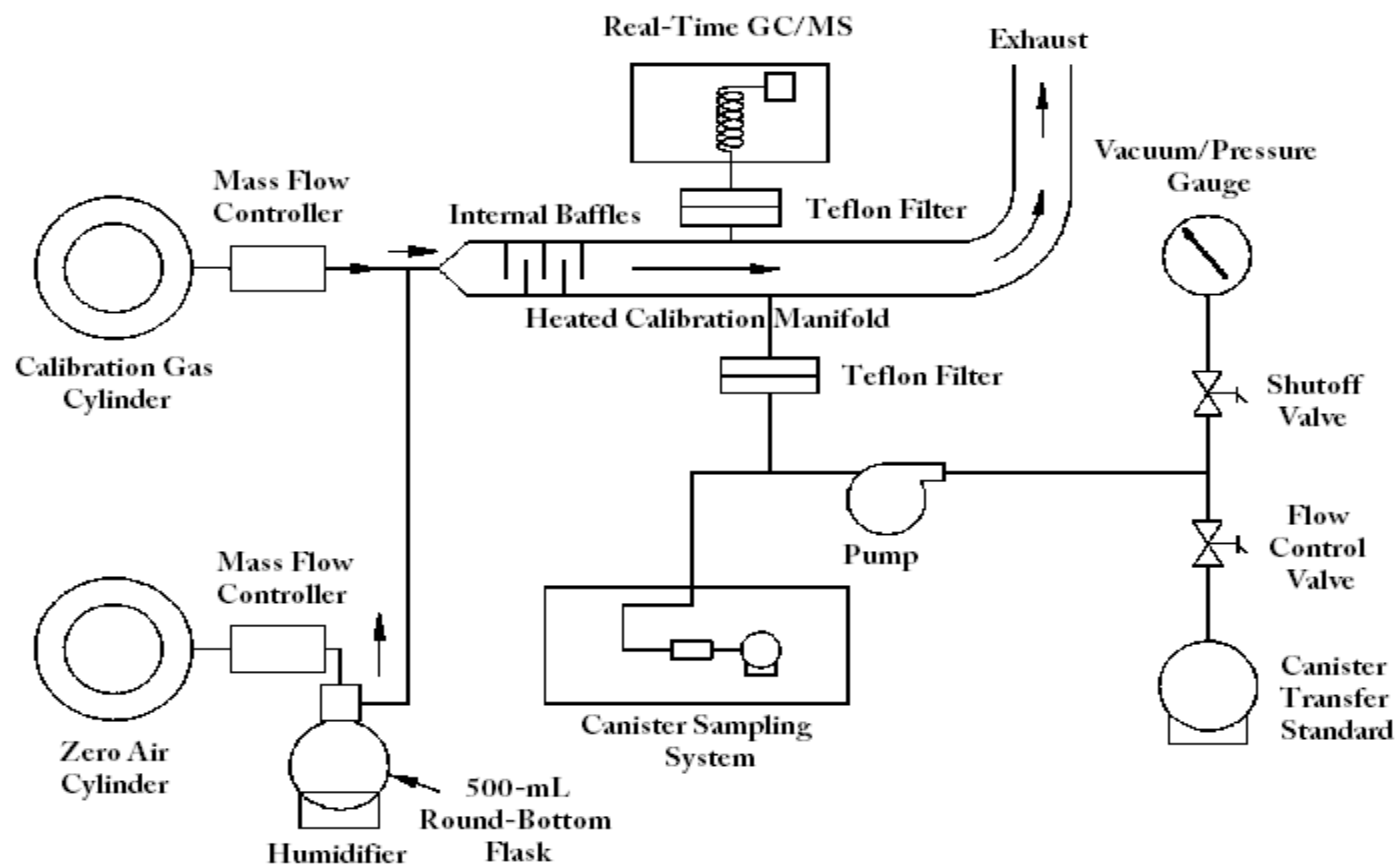


Figure 3. Schematic of a dynamic calibration system, which can be used for testing cleanliness of a sampling train (Section 9.1.5), calibrating the GC/MS (Section 11.1 and SOP V6), or preparing canister transfer standards through dynamic dilution (SOP V6). Adopted from USEPA Compendium Method TO 14A.

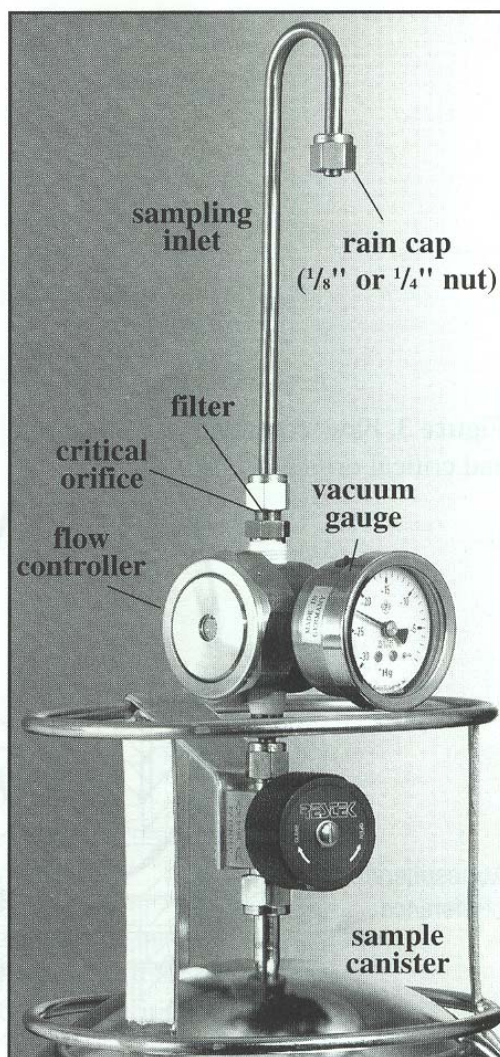


Figure 4. Proper assembly and attachment to a canister of a sampling train. Source: RESTEK Corporation (Reference 12.5).

**MEASUREMENT OF WIND WITH THE RM YOUNG
MODEL 81000 3-DIMENSIONAL SONIC ANEMOMETER**

Standard Operating Procedure (SOP) W2

**MEASUREMENT OF WIND WITH THE
RM YOUNG MODEL 81000 3-DIMENSIONAL SONIC ANEMOMETER
Standard Operating Procedure (SOP) W2**

**Prepared by
Richard H. Grant**

**Reviewed by
Albert J. Heber and Bill W. Bogan**

**Effective Date: November 6, 2006
Rev. 1.0 Effective Date: February 23, 2008**

**PURDUE APPLIED METEOROLOGY LABORATORY (PAML)
Agronomy, Purdue University
West Lafayette, IN 47907**

Table of Contents

	Page
1. Scope and Applicability	3
2. Summary of Method	3
3. Definitions	3
4. Health and Safety	3
5. Cautions	4
6. Interferences	4
7. Personnel Qualifications	4
8. Equipment and Supplies	4
9. Procedures	4
10. Data and Records Management	Error! Bookmark not defined.
11. Quality Control and Quality Assurance	14
12. References	16
13. Contact Information	17
APPENDIX A. Audit Form	19
APPENDIX B. Field Intercomparison Form	20

1. Scope and Applicability

- 1.1. The measurement of the directional components of wind is used in the gaseous emissions measurement methodologies.
 - 1.1.1. Statistical values of instantaneous wind components and the virtual temperature are used in the backward Lagrangian Stochastic method of emission measurement (SOP O1).
 - 1.1.2. Statistical values of instantaneous horizontal wind components are used in the radial plume method of emission measurement (SOP O2).
- 1.2. The difference in time required for sound to travel through the moving air is a fast response method of measuring each directional component of the wind.
- 1.3. The detection limit of the method described in this SOP is 0.05 m/s.
- 1.4. The range of wind speeds which can be measured under this SOP is 0.05 to 40 m/s.
- 1.5. For wind speed, the manufacturer-reported sensor accuracy is 0.02 m/s, while the manufacturer-reported sensor precision (rms) is 0.05 m/s for speeds of 0 to 5 m/s, 1% of reading for speeds from 5 to 30 m/s and 3% of reading for speeds from 30 to 40 m/s.
- 1.6. This procedure applies only to the RM Young Model 81000 Ultrasonic Anemometer.

2. Summary of Method

The RM Young Model 81000 measures three-dimensional wind velocity and speed of sound based on the transit time of ultrasonic acoustic signals at 4Hz to 32 Hz. Sonic temperature is derived from speed of sound which is corrected for crosswind effects. Measurement data are available as serial output using RS-232 or RS-485 connections. Operating parameters may be edited via simplified menus using an ordinary serial communication program like HyperTerminal®. All parameters are stored in non-volatile memory. Superior environmental resistance is achieved by using UV stabilized thermoplastic, stainless steel, and anodized aluminum components.

3. Definitions

COM port	A serial communications port of a computer
HyperTerminal®	A Windows® program that provides communication through a serial communications port of a computer
PAAQL	Purdue Agricultural Air Quality Laboratory
PAML	Purdue Applied Meteorology Laboratory
QAPP	Quality Assurance Project Plan
RS-232	A serial communications protocol
RS-485	A serial communications protocol

4. Health and Safety

- 4.1. The sensor has electrical components, and proper care should be used as with all electrical equipment.

5. Cautions

- 5.1. The sensor is acoustical and therefore cannot be dropped without damage.
- 5.2. Be sure that the sensor is properly grounded.
- 5.3. Tilt of sensor induces an error in the measurement of approximately 6% in the correlations of wind deviations and 3% in the derived friction velocity with 1° tilt.

6. Interferences

- 6.1. Wakes from obstructions such as buildings, storage piles, trees and other vegetation which are closer than ten times the sensor height.
 - 6.1.1. Incorrect siting of the anemometer will cause greater uncertainties in the data due to failure of assumptions of homogeneity and stationarity.
- 6.2. Sensor response under high winds can result in extreme values.
- 6.3. The sensor does not respond under conditions of accumulated freezing rain, heavy frost and snow on the transducers.
- 6.4. Wake flow around support struts reduces sensor performance.

7. Personnel Qualifications

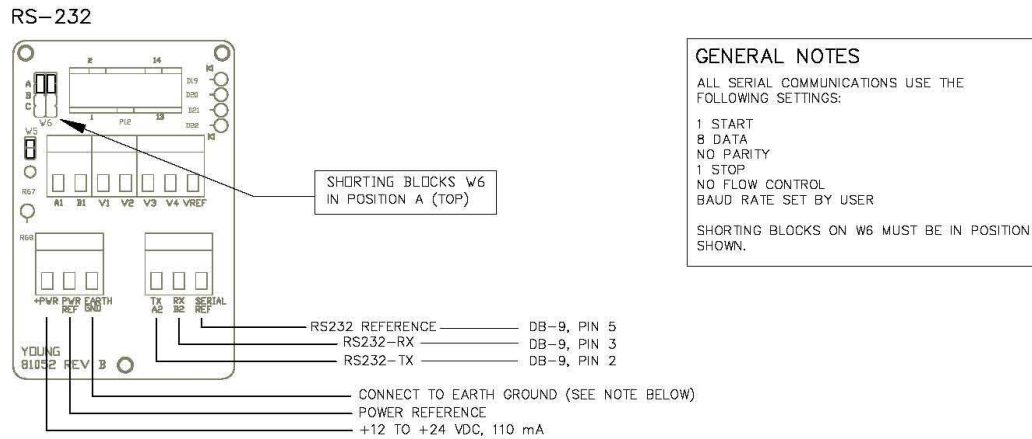
- 7.1. Read and understand the sensor instruction manual and this SOP before operating this sensor.

8. Equipment and Supplies

- 8.1. 3-D sonic anemometer
- 8.2. #2 common head screwdriver
- 8.3. 1/8" slotted screwdriver
- 8.4. Anechoic box
- 8.5. Laptop computer
- 8.6. DB9 modem cable
- 8.7. Labview software
- 8.8. XStream® RS-232/485 RF Modem
- 8.9. Sonic anemometer intercomparison stand

9. Procedures

- 9.1. Instrument acceptance
 - 9.1.1. Carefully unpack the unit and inspect for physical damage. Report any damage to the shipper. The sensor arrives fully calibrated and ready to use.
 - 9.1.2. Remove junction box cover. Connect power and signal wires to terminals as in the wiring diagram below. Connect serial cable to computer COM port.
 - 9.1.2.1. Supply power must be in the range of 12 to 24 VDC **at the sensor junction box terminals** in order for the sensor to operate properly.



9.1.3. Start HyperTerminal® on the computer with baud rate at 38400 and flow control set to NONE.

9.1.4. Apply power to the 81000 sensor. There will be a 4-s delay for initialization; then, the unit will begin to output data at 4 Hz using the following format: speed (m/s), azimuth (deg), elevation (deg), speed-of-sound (m/s), and sonic-temperature (°C). Verify that all values are present on the display. Typical output is:

0.00	0.0	0.0	346.70	25.14
0.00	0.0	0.0	346.68	25.11
0.00	0.0	0.0	346.76	25.25
0.00	0.0	0.0	346.80	25.30
0.00	0.0	0.0	346.82	25.35

Wind below the 0.05 m/s detection limit is output as 0.00 m/s. Azimuth may be any value from 0.0 to 359.9 degrees. When wind speed is below threshold, azimuth output is maintained at the last value read before wind speed went below threshold.

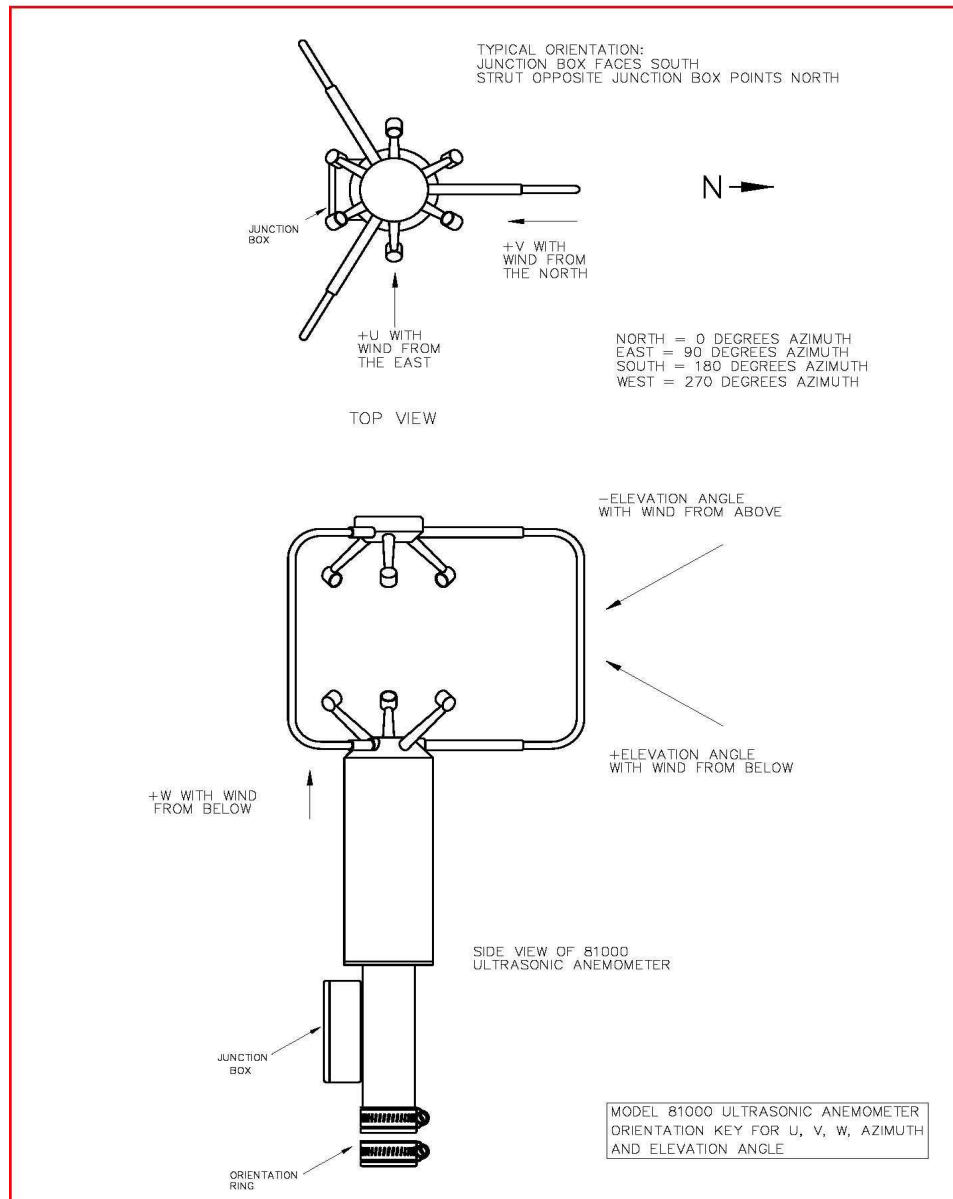
Elevation remains zero until threshold is exceeded. Speed of sound ranges from 300 m/s to 360 m/s, depending on temperature. At 20°C, the value is ~ 344 m/s. Sonic temperature may be compared to a standard Celsius thermometer and should agree within ±2°C. If values appear questionable or any value is not displayed, remove power and check all wiring connections. If the problem cannot be corrected, contact service support (Section 13.1).

9.1.5. Verify sensor response by gently blowing through the measuring section. Wind from the north side (marked “N”), should produce a positive SPEED response and an AZIMUTH display corresponding to North (i.e.: values around 359.9 or 0.0). Wind from the opposite direction should produce AZIMUTH values indicating south, (around 180) and so forth. Downward wind produces negative ELEVATION values, upward wind produces positive values.

9.2. Instrument installation

9.2.1. Mount the sensor securely on top of a mast or tower such that it will not twist, rotate, or sway, according to SOP U5.

9.2.2. Mount the sensor with the following orientation:

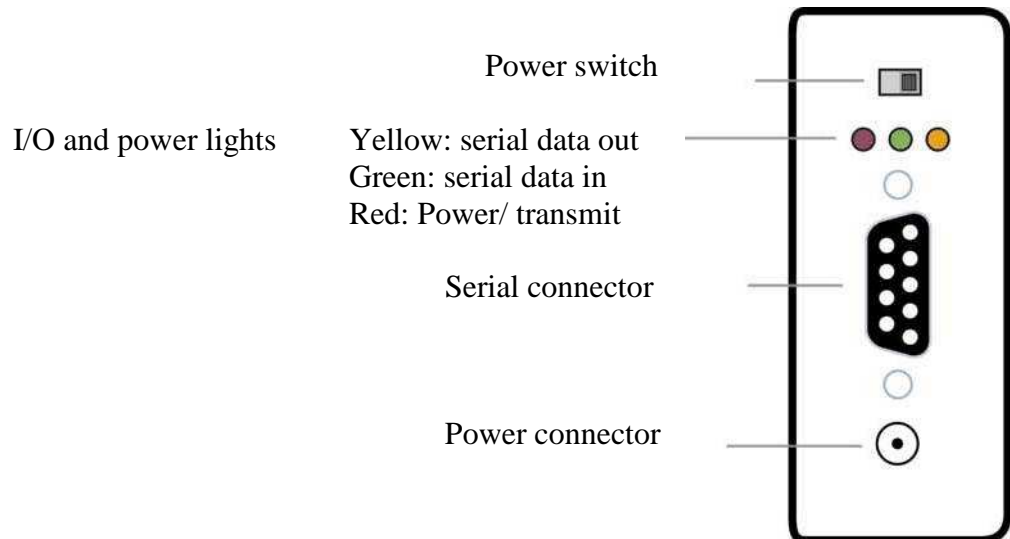


9.2.3. Attach earth ground for proper operation.

9.3. Instrument operation

- 9.3.1. Connect a serial cable from the XStream RS-232/485 RF Modem to the COM port of a laptop computer.
- 9.3.2. Connect antenna to RF Modem.
- 9.3.3. Provide power to radio modem by connecting power cable to power connector and switching on power switch (See figure below)

9.3.4. Verify power to radio modem by checking red light on modem indicated below:



9.3.4.1. Using X-CTU® program on the laptop computer, verify configuration of Modem (8 data bits, 1 stop bit, no parity, 19200 baud), in accordance with SOP U5.

9.3.5. Connect the serial cable from XStream RS-232/485 RF Modem serial output connector (See figure in Section 9.3.5) to the anemometer, according to figure in Section 9.1.2.

9.3.5.1. Test communication link to the computer in the trailer.

9.3.6. Establish communications with the sensor, according to Sections 9.1.3 and 9.1.4.

9.3.7. Provide power to sensor, and wait 4-s for initialization.

9.3.8. Using the HyperTerminal® window, send the ESC character (ASCII 27) three times in quick succession. This takes the unit out of OPERATE mode and causes the COMMAND MENU to appear:

```
COMMANDS (VERSION 6.6.07)
```

```
-----
```

```
R) REPORT
```

```
S) SETUP
```

```
X) EXIT TO OPERATE MODE
```

9.3.9. Go to SETUP. The SETUP menu appears as follows:

```
SET PARAMETERS
-----
A) AVERAGING
B) BAUD
E) ERROR HANDLING
N) SCALING MULT
O) OUTPUT RATE
P) POLL CHARACTER (ADDR)
S) SER OUT FORMAT
T) THRESHOLD
U) UNITS
V) V OUT FORMAT
W) WAKE CORRECTION
X) EXIT TO MAIN MENU
```

9.3.9.1. Set AVERAGING at 0.

9.3.9.2. Set ERROR HANDLING to "INCLUDE INVALID DATA".

9.3.9.3. Set OUTPUT RATE at 16-Hz

9.3.9.4. Set SER OUT FORMAT as "CUSTOM". The following appears:

```
CURRENT SERIAL OUTPUT FORMAT:
[   ]
CONSTRUCT AN OUTPUT FORMAT BY SELECTING FROM THE LIST BELOW.
ELEMENTS MAY BE IN ANY ORDER. REFER TO MANUAL FOR DETAILS.
5) UVW
6) 2D SPEED
7) 3D SPEED
8) AZIMUTH
9) ELEVATION
A) SOS
B) Ts
E) ERR CODE
V) INTERNAL VOLTAGE
```

ENTER CUSTOM STRING (12 CHARACTERS MAX):

9.3.9.4.1. Enter Custom string as "5BE".

9.3.9.5. Set WAKE CORRECTION to "YES".

9.3.9.6. Exit to main menu and return to OPERATE mode.

9.3.9.7. Locate the appropriate input serial channel on the computer in trailer (SOP U4) and evaluate output for reasonable values.

9.3.9.8. Check output of sensors on the instrument trailer computer.

9.3.9.8.1. If output is not providing three wind vectors, a temperature and an error code, in accordance with the manual, re-enter setup menu and correct problem, starting anew at Section 9.3.5.

9.4. Instrument calibration

- 9.4.1. The sensor response under constant wind is calibrated at the factory.
- 9.4.2. The sensor cannot be calibrated for turbulence components.
- 9.4.3. Standard intercomparison: Compare the output of all three in-use sensors with that of three unused sensors (Standard Triad). This is done after every 6 mos.
 - 9.4.3.1. Locate the sensors to minimize wind interference given prevailing winds.
 - 9.4.3.2. Conduct measurements with the sensors for a period of 24-48 h, so that a variety of atmospheric conditions are included. Then, reverse the locations of the sensors and sample for another 24-48 h.
 - 9.4.3.3. Compare the measurements obtained from the three sensors of the Standard Triad to ensure that the mean wind speed agrees to within 0.2 m/s. If the sensors agree within this amount, then use the average of all 3 sensors of the Triad as the standard. If one of the sensors lies outside this range, then use the average of the two remaining sensors as the standard.
 - 9.4.3.3.1. Troubleshoot any Standard sensor that is out of agreement with other sensors, and resolve problem (Section 9.5).
 - 9.4.3.4. Compare the mean wind speed from each used sensor with the standard set obtained above. If the mean difference is less than 0.2 m/s, then the used sensor being compared is in good working order. If the mean difference is greater than 0.2 m/s, then the used sensor being compared is defective
 - 9.4.3.4.1. Troubleshoot defective sensor and resolve problem (Section 9.5).
- 9.4.4. In-field intercomparison: Compare the sensor with two identical units which are in routine use.
 - 9.4.4.1. Locate all sensors next to each other on the sonic anemometer intercomparison stand. Orient the stand so that it is perpendicular to the wind, to minimize interference among the instruments.
 - 9.4.4.2. Conduct measurements with all sensors for a one-hour period. Record the mean wind speeds for the three sensors over this period on the Field Intercomparison Form (Appendix B).
 - 9.4.4.3. Determine the three paired differences between the sensors' measurements.
 - 9.4.4.3.1. If all paired differences are less than 0.2 m/s, then the sensors are in good working order.
 - 9.4.4.3.2. If one of the sensor's measurement differs by more than 0.2 m/s from the other sensors' measurements, then conduct tests for another one-hour period. If the difference remains greater than 0.2 m/s, then the sensor is defective, and must be returned to the factory for repair or replacement. Otherwise, if the difference in the second hour is less than 0.2 m/s, then the differences in the first hour were likely due to unsteady wind conditions.
 - 9.4.4.3.2.1. If a sensor fails this test, inspect all measurements made since the sensor last passed to determine the time of failure. Invalidate measurements made after that time.
 - 9.4.4.3.2.2. Contact PM for sensor replacement.

9.5. Troubleshooting

9.5.1. If no measurements are reported at computer:

9.5.1.1. Check for obstructions in the acoustic path. Invalid measurements occur when the acoustic path of the sonic signal is blocked or internal circuits fail.

9.5.1.1.1. Acoustic blockage may be caused by rain drops, ice, snow, or other debris, and is indicated by the output error code (see Section 11).

9.5.1.2. Check communications.

9.5.1.2.1. Verify communications link between radio modems (Yellow and Green lights intermittently lit during communications) on modems; (See figure in Section 9.3.3).

9.5.1.2.2. Verify firm connections for power, signal, and antenna for serial communications.

9.5.1.2.2.1. Correct as needed.

9.5.1.2.3. Verify power at modems in trailer and at anemometer, as indicated by a lit red light on the modems (See figure in Section 9.3.3).

9.5.1.2.3.1. If not within acceptable range, check continuity of wires and tightness of cable connections and correct as needed.

9.5.1.2.3.1.1. If all connections are tight and of good continuity, send sensor to manufacturer for repair and/or replacement of the appropriate modem.

9.5.1.2.4. Verify Destination Address of radio modems (SOP W6), and correct as needed.

9.5.1.2.5. Check that the correct baud rate is selected (See Section 9.1.3) and correct as needed.

9.5.1.3. Sensor troubleshooting

9.5.1.3.1. Verify power at anemometer by voltage measurement with multimeter: Voltage should be 12 to 24 VDC **at the sensor junction box terminals** (See figure in Section 9.1.2).

9.5.1.3.1.1. If voltage is not within acceptable range, check continuity of wires and tightness of cable connections.

9.5.1.3.1.1.1. Correct as needed.

9.5.1.3.1.1.2. If all connections are tight and of good continuity, send sensor to manufacturer for repair or replacement.

9.6. Data acquisition, calculations & data reduction requirements

9.6.1. Data are collected on the LAN server (SOP D1).

9.6.2. Corrections for wake effects are internally conducted.

9.6.3. Identify spikes in 16-Hz values of U , V , W , T using 100-s mean filter and dynamically calculated standard deviation.

9.6.3.1. Calculations are made by a computer program on the LAN server (SOP D1).

9.6.3.2. Spikes are defined as data points with values more than 3 standard deviations away from running mean.

9.6.3.2.1. Spike data are omitted.

9.6.3.3. Count spikes and calculate their mean value.

9.6.3.4. Inspect and flag data falling outside of acceptable limits based on spike counts.

9.6.3.4.1. Write to a data file the spike count, as well as the mean value of spikes, over a 5-min interval.

- 9.6.3.4.2. If spike count is greater than 160 for any variable, then the 5-min measurement period for that variable is flagged and investigated.
- 9.6.3.4.2.1. Do not calculate the derived variables in Sections 9.6.4 and 9.6.5 using any invalidated variables as dependencies.
- 9.6.3.4.3. If spikes count is less than 160 for a variable, then that variable passes the spike test.
- 9.6.3.4.3.1. Store instantaneous U , V , W , T and corresponding qc flags (See table in Section 11.4.1) in temporary data file.
- 9.6.3.4.3.2. Store 16-Hz sampling of instantaneous values of wind components u' and w' from 2 m and 16 m anemometers over the period from 12:00 PM to 12:30 PM in a data file (for spectral analysis on LAN server).
- [NOTE: There is no correction for tilt of the sensor since inhomogeneities of the surface type (lagoon vs. land surrounding lagoon) and terrain elevation (lagoon berm) create streamlines assumed to be variable with wind direction].
- 9.6.4. Mean horizontal winds derived from 2-s periods of 16-Hz sampling of instantaneous wind components u' and v' .
- 9.6.4.1. Computations are made by computer program on the LAN server (SOP D1).
- 9.6.4.2. Compute the means of U , V (\bar{U} , \bar{V}). Computed values are used in the radial plume method of emission measurement (SOP O2).
- 9.6.5. Turbulence statistics derived from 5-min periods of 16-Hz sampling of instantaneous wind components u' , v' , w' and T' .
- 9.6.5.1. Computations are made by computer program on the LAN server (SOP D1).
- 9.6.5.2. Compute the means of U , V , W , and T (\bar{U} , \bar{V} , \bar{W} , \bar{T}). Computed values are used in the quality assurance routines and the backward Lagrangian Stochastic emissions measurement (SOP O1).
- 9.6.5.3. Compute the variance of the three orthogonal wind components σ_u^2 , σ_v^2 , σ_w^2 and σ_T^2 ($\overline{u'u'}$, $\overline{v'v'}$, $\overline{w'w'}$, $\overline{T'T'}$). Computed values are used in the Quality Control and Assurance checks (Section 11) and the backward Lagrangian Stochastic emissions measurement (SOP O1).
- 9.6.5.4. Compute averaged correlations of instantaneous deviations in wind components u' , v' , w' and the temperature T' derived from the speed of sound: $\overline{u'w'}$, $\overline{u'v'}$, $\overline{v'w'}$, $\overline{u'T'}$, $\overline{w'T'}$, $\overline{v'T'}$. Computed values are used in the Quality Control and Assurance checks (Section 11) and the backward Lagrangian Stochastic emissions measurement (SOP O1).
- 9.6.6. Mean horizontal winds derived from 5 and 30-min periods of 16-Hz sampling of instantaneous wind components u' and v' .
- 9.6.6.1. Computations are made by computer program at PAML.
- 9.6.6.2. Computed values are used to characterize wind conditions.
- 9.6.6.3. Compute mean vector wind speed (\bar{S}) according to the following:

$$\bar{S} = \sqrt{\bar{U}^2 + \bar{V}^2}$$

9.6.6.4. Compute mean wind direction ($\bar{\Theta}$) according to:

$$\bar{\Theta} = \left\{ \begin{array}{ll} 0^\circ & \text{if } \bar{U} = 0 \text{ and } \bar{V} > 0 \\ 180^\circ & \text{if } \bar{U} = 0 \text{ and } \bar{V} < 0 \\ -\tan^{-1}\left(\frac{\bar{V}}{\bar{U}}\right) + 90^\circ & \text{if } \bar{U} > 0 \\ -\tan^{-1}\left(\frac{\bar{V}}{\bar{U}}\right) + 270^\circ & \text{if } \bar{U} < 0 \end{array} \right\}$$

The convention for the sonic anemometer output is that U is positive for wind from the east, and V is positive for wind from the north. The directions obtained from the above formula are 0° for wind from the north, 90° for wind from the east, and so on.

9.6.7. Turbulence statistics derived from 5 and 30-min periods of 16-Hz sampling of instantaneous wind components u' , v' , w' and virtual temperature T' .

9.6.7.1. Computations are made by computer program at PAML.

9.6.7.2. Re-compute LAN-computed variances σ_u^2 , σ_v^2 , and σ_w^2

and correlations $\overline{u'w'}$, $\overline{u'v'}$, $\overline{v'w'}$, $\overline{u'T'}$, $\overline{w'T'}$, $\overline{v'T'}$ with rotated coordinates so that mean wind is in the u direction.

9.6.7.2.1. Rotated values are used in the stationarity and homogeneity quality assurance tests (Sections 11.3.2, 11.3.3)

9.6.7.3. Compute the friction velocity (u_*):

$$u_* = \sqrt[4]{\overline{u'w'^2} + \overline{v'w'^2}} \quad (\text{Flesch et al. 2005})$$

9.6.7.3.1. Computed values are used to characterize wind conditions, and to qualify backward Lagrangian Stochastic emission measurements (SOP O1).

9.6.7.4. Compute the characteristic temperature:

$$T_* = \overline{w'T'} / u_* \quad (\text{Kaimal and Finnigan 1994})$$

9.6.7.4.1. Computed values are used to characterize wind conditions.

9.6.7.5. Compute the Obukov length (L):

$$z/L = \frac{-kzg\overline{w'T'}}{\overline{T}u_*^3} \quad (\text{Flesch et al. 2005})$$

Where:

z	=	Sensor height (m)
k	=	von Karman's constant (0.4)
g	=	Acceleration due to gravity (9.8 m/s)

9.6.7.5.1. Computed values are used to characterize wind conditions and qualify backward Lagrangian Stochastic emission measurements (SOP O1).

9.6.7.6. The program estimates the roughness length (z_o) as the average value of the three heights of wind measurement, each calculated as:

$$z_o = \frac{(z-d)}{e^{kU/u_* - \psi_m}} \quad (\text{Flesch et al. 2005})$$

Where (Flesch et al. 2004, Businger et al. 1971, Paulson 1970):

$$\begin{aligned} \psi_M &= 4.7 (z-d)/L \text{ for } z/L > 0.0001 \\ \psi_M &= 0 \text{ for } 0.0001 > z/L > 0.0001 \\ \psi_M &= -2 \ln[(1+x)/2] - \ln(1+x^2)/2 + 2 \tan^{-1} x - \pi/2 \text{ and} \\ X &= (1-15(z-d)/L)^{1/4} \text{ for } z/L > 0.0001 \end{aligned}$$

9.6.7.6.1. Computed values are used to characterize wind conditions.

9.6.7.7. Estimate dissipation rate ε :

$$\varepsilon = \frac{u_*^3}{kz} \varphi_\varepsilon \quad (\text{Flesch et al. 2004})$$

Where:

$$\varphi_\varepsilon = 1 + 5z/L \quad \text{for } L > 0$$

$$\varphi_\varepsilon = \frac{b_w^4 (1 - 3z/L)^{1.33} + 1}{(b_w^4 + 1)(1 - 3z/L)^{0.33} (1 - 6z/L)^{0.25}} \quad \text{for } L < 0$$

9.6.7.7.1. Computed values are used to characterize wind conditions.

9.6.8. Write values of \overline{U} , \overline{V} , \overline{W} , \overline{T} , \overline{S} , $\overline{\Theta}$, $\sigma_u^2(\overline{u'u'})$, $\sigma_v^2(\overline{v'v'})$, $\sigma_w^2(\overline{w'w'})$, u_* , T_* , z/L , z_o , ε and corresponding qc flags (Table 1) to data file

9.7. Computer hardware & software

9.7.1. Using electrical wires, attach sensor serial port to an XStream® RS-232/485 RF Modem.

9.7.2. A computer in the instrumentation trailer will receive the data using an identically addressed XStream® RS-232/485 RF Modem on a COM port.

9.7.3. Computer will record and process the data, and store it using LABView software.

10. Data and Records Management

10.1. Manage data according to SOP D1.

10.2. Document all data and information checked in the field on field data sheets and in site logbooks with permanent ink, or in electronic notes.

10.3. Overstrike errors in writing with a single line; date and initial all such corrections.

11. Quality Control and Quality Assurance

11.1. Physical inspection

11.1.1. Inspect sensor support arms for dents or deep scratches in the metal.

11.1.1.1. If present, conduct an in-field intercomparison (Section 9.4.3).

11.2. Zero check

11.2.1. Enclose sensor within an anechoic box and check for non-zero values on all wind components (similar to ASTM 2001).

11.2.1.1. Record sensor biases in the U , V , and W components on an audit form similar to the one in Appendix A of this SOP.

11.2.1.2. If sensor does not indicate values of less than 0.2 m/s in all three components, then send it back to the factory for repair or replacement.

11.3. Path obstruction check

11.3.1. The instrument's internal processing determines individual invalid measurements due to a path obstruction (e.g. debris, dew, mist, ice, or snow) preventing or radically altering acoustic transit time measurement. Instrument output includes invalid data and an error code, the sum of values for the following conditions:

2	Zero samples on at least one acoustic path
4	Path A speed < -50 m/s
8	Path A speed > +50 m/s
16	Path B speed < -50 m/s
32	Path B speed > +50 m/s
64	Path C speed < -50 m/s
128	Path C speed > +50 m/s

11.3.1.1. If less than 10% of the sampling interval has non-zero error codes, the measurement period is considered valid and computation on the valid data beginning at section 9.6 will be conducted.

11.3.1.2. If greater than 10% of the sampling interval has a non-zero error code, the measurement period is given an error code (Table 1).

11.3.2. Stationarity check Determine stationarity in the time series by comparing 5-min covariances of u' and w' and w' and T_v' (Section 9.6.6.2) with the 30-min covariance (Section 9.6.5) where i and j are different variables:

$$\sum \left[\left(\overline{u'_i u'_j} \right)_{5 \text{ min}} - \left(\overline{u'_i u'_j} \right)_{30 \text{ min}} \right] / \left(\overline{u'_i u'_j} \right)_{30 \text{ min}} \quad (\text{Foken and Wichura 1996})$$

where the 5-min periods included in the sum are sequential within the 30-min interval. Computations are done by computers at PAML.

11.3.2.1. If greater than 0.30 with the inclusion of any 5-min period to the sum, the 5-min period of data is given a QA/QC error code (Table 1).

11.3.3. Homogeneity check: Given values computed according to Section 9.6.6.2, check for homogeneity of turbulence during 30-min sample periods (six sequential 5-min periods) by comparing the theoretically derived and measured integral turbulence

scales of w' , u' and T' (u_* and T_*) to the standard deviation of w' and T' . Computations are done by computers at PAML.

11.3.3.1. The theoretically-derived values are computed according to (Foken and Wichura 1996):

z/L	<-1	$-1 \dots -0.0625$	$-0.0625 \dots <0$	≥ 0
$\frac{\sigma_w}{u_*}$	$2.00(-z/L)^{1/6}$	$2.00(-z/L)^{1/8}$	1.41	1.25
$\frac{\sigma_u}{u_*}$	$2.83(-z/L)^{1/6}$	$2.83(-z/L)^{1/8}$	1.99	2.4
$\frac{\sigma_T}{T_*}$	$1.00(-z/L)^{1/3}$	$1.00(-z/L)^{1/4}$	$0.50(-z/L)^{1/2}$	

11.3.3.2. Compute the differences between theoretical and measured values as:

$$\sum \left[\frac{\sigma_w}{u_*} - \left(\frac{\sigma_w}{u_*} \right)_{theory} \right] / \left(\frac{\sigma_w}{u_*} \right)_{theory}$$

$$\sum \left[\frac{\sigma_u}{u_*} - \left(\frac{\sigma_u}{u_*} \right)_{theory} \right] / \left(\frac{\sigma_u}{u_*} \right)_{theory}$$

$$\sum \left[\frac{\sigma_T}{T_*} - \left(\frac{\sigma_T}{T_*} \right)_{theory} \right] / \left(\frac{\sigma_T}{T_*} \right)_{theory}$$

11.3.3.2.1. If the value of any of these is greater than 0.30, the 30-min period is given a QA/QC error code (Table1).

11.4. Turbulence structure check: Conduct spectral turbulence checks of the 16-Hz sampling of instantaneous wind components u' and w' collected over an approximately 30-min period between 12:00 AM and 12:30 PM. Computations are done by computers at PAML.

11.4.1. Store instantaneous values of wind components u' , v' and w' from all anemometers in a data file for processing at PAML.

11.4.2. Calculate spectral statistics of u' and w' :

11.4.3. Calculate the spectral density function of the first 32,384 values at the all heights by fast Fourier transform.

11.4.4. Using u_* calculated for the 30-min period, calculate the spectral density function normalized to u_*^2 and plot as

$$fS(f)/u_*^2 \text{ by } f \text{ for both measurement heights.}$$

11.4.5. Compare shape of spectra and evaluate similarity.

11.4.5.1. If more than one peak in the spectra for either height, flag period values as suspect (Table 1).

11.4.5.2. If the peak spectral density in the 16-m height spectra is at a higher frequency than that for the 2-m height, flag period values as suspect (Table 1).

12. References

- 12.1. ANL. 2006. Data processing and analysis for ABLE surface characterization, eddy correlation flux measurement system. Argonne National Laboratory Atmospheric Boundary Layer Experiment. Online at <http://www.atmos.anl.gov/ABLE/EC.pdf>. Accessed 1-31-2006.
- 12.2. ASTM 2001. Standard Test Method for Determining the Performance of a Sonic Anemometer/Thermometer. American Society for Testing and Materials, Philadelphia, PA, No. D6011-96.
- 12.3. Baxter, RA, DL Yoho, KR Durkee, 2003. Quality assurance audit program for surface and upper-air meteorological measurements in the south coast air basin of California. 12th Symposium of Meteorological Observations and Instruments, Long Beach, CA.
- 12.4. Businger, JA, JC Wyngaard, Y Izumi, and EF Bradley. 2001. Flux-profile relationships in the atmospheric surface layer. *J. Atmos. Sci.* 28, 181-189.
- 12.5. Flesch, TK, JD Wilson, LA Harper, BP Crenna, and RR Sharpe. 2004. Deducing ground-to-air emissions from observed trace gas concentrations: A field trial. *J. Appl. Meteorol.* 43, 487-502.
- 12.6. Flesch, TK, JD Wilson, LA Harper, and BP Crenna. 2005. Estimating gas emissions from a farm with an inverse-dispersion technique. *Atmos. Environ.* 39, 4863-4874.
- 12.7. Foken, Th., B. Wichura. 1996. Tools for quality assessment of surface-based flux measurements. *Agric. Forest Meteorol.* 78, 83-105.
- 12.8. Kaimal, JC and JJ Finnigan, 1994. *Atmospheric Boundary Layer Flows: Their Structure and Measurement*. Oxford University Press, Inc., New York, NY.
- 12.9. X-Stream modem manual. 2005. XStream-PKG-R RS-232/485 RF Modem. Product Manual v4.2B. Maxstream Inc, Lindon, UT.
- 12.10. Panofsky, HA and JA Dutton. 1984. *Atmospheric Turbulence*. Wiley Interscience Press, NY.
- 12.11. Paulson, CA. 1970. The mathematical representation of wind speed and temperature profiles in the unstable atmospheric surface layer. *J. Appl. Meteor.* 9, 857-861.
- 12.12. Model 81000 Manual. 2005. Manual for Model 81000 Ultrasonic anemometer, Rev C031405. R.M. Young Co., Traverse City, MI. 9p.
- 12.13. SOP D1. 2006. Management of Open-source, Weather, and Lagoon-characterization Data. Standard Operating Procedure D1. Purdue Applied Meteorology Lab.
- 12.14. SOP O1. 2006. Emissions Estimation Using the Thunder Beach Scientific Windtrax Backward Lagrangian Stochastic Model. Standard Operating Procedure O1. Purdue Applied Meteorology Lab.
- 12.15. SOP O2. 2006. Emissions Estimation Using the ARCADIS Radial Plume Mapping Model. Standard Operating Procedure O2. Purdue Applied Meteorology Lab.

- 12.16. SOP U5. 2006. Installation of Open-source Measurement Equipment. Standard Operating Procedure U5. Purdue Applied Meteorology Lab.
- 12.17. SOP W6. 2006. Establishment, Data Acquisition and Control of Weather and Lagoon Characterization Hardware. Standard Operating Procedure W6. Purdue Applied Meteorology Lab.
- 12.18. USEPA. 1995. Quality Assurance Handbook for Air Pollution Measurement Systems, Volume IV- Meteorological Measurements EPA-600/R-94-038d, U.S. Environmental Protection Agency, Research Triangle Park, NC.
- 12.19. USEPA. 2000. Meteorological Monitoring Guidance for Regulatory Modeling Applications. EPA-454/R-99-005, U.S. Environmental Protection Agency, Research Triangle Park, NC
- 12.20. Wilzak, JM, SP Oncley, SA Stage. 2001. Sonic anemometer tile correction algorithms. Boundary-Layer Meteorol. 99, 127-150.

13. Contact Information

13.1. Anemometer manufacturer:

R. M. Young Company, 2801 Aero Park Drive, Traverse City, MI, 49686, USA,

email: met.sales@youngusa.com

Customer support and repair: 231-946-3980, Fax 231-946-4772

13.2. Modem manufacturer:

Maxstream, Inc., 355 South 520 West, Suite 180, Lindon, UT 84042

Phone: (801) 765-9885

Fax: (801) 765-9895

email: rf-xperts@maxstream.net

Table 1. Data QA/QC conditions.

Individual measurements

Condition
Value not suspect
Value missing (>10% of records in 5 min period)
Value below minimum or above maximum or value is +/- infinity
Spike count >160
Value has variance >10 m ² /s ² (u, v, w) or 10 C ² (T)
> 10% of sampling interval has path obstruction

Combinational measurements

u'w' not stationary (30-min period)
w'T' not stationary (30-min period)
σ_u inhomogeneous turbulence in 5-min period
σ_w inhomogeneous turbulence in 5-min period
σ_T inhomogeneous turbulence in 5-min period

**MEASUREMENT OF WETNESS WITH THE CAMPBELL
SCIENTIFIC MODEL RESISTANCE GRID**

Standard Operating Procedure (SOP) W3

**MEASUREMENT OF WETNESS WITH THE CAMPBELL
SCIENTIFIC MODEL RESISTANCE GRID
Standard Operating Procedure (SOP) W3**

Prepared by

Juan Carlos Ramirez and Richard H. Grant

Reviewed by

Bill W. Bogan and Albert J. Heber

Effective Date: November 6, 2006

Rev. 1.0 Effective Date: February 6, 2008

PURDUE APPLIED METEOROLOGY LABORATORY (PAML)

Agronomy, Purdue University

West Lafayette, IN 47907

Table of Contents

	Page
1. Scope and Applicability.....	3
2. Summary of Method.....	3
3. Definitions.....	3
4. Cautions.....	3
5. Interferences.....	4
6. Personnel Qualifications	4
7. Equipment and Supplies	4
8. Procedures.....	4
9. Data and Records Management	6
10. Quality Control and Quality Assurance.....	6
11. References.....	7
12. Contact Information.....	7
Appendix A. Calibration Check Form.....	8

1. Scope and Applicability

- 1.1. Electrical resistance grids are useful for the determination of surface wetness.
- 1.2. Surface wetness measurements indicate precipitation, an important weather variable.
- 1.3. This procedure applies only to the Campbell Scientific Model 237 Leaf Wetness Sensor Resistance Grid, to be used coated with white latex ceiling house paint.
- 1.4. This device is used as a wet/dry sensor in this application. Sensor readings in between wet and dry are simply interpreted as “Conditions other than wet or dry”.
 - 1.4.1. If measuring resistance with a multimeter, connect the leads to the red and black sensor leads. The sensor lead has a built-in 100,000- Ω , resistor in series with the sensor that must be included in any resistance calculations. See R1 in Fig. 1 of the Model 237 manual. The resistance (including the 100,000- Ω resistor) will be approximately 120,000 Ω – 500,000 Ω when wet, and above 3,100,000 Ω when dry.

2. Summary of Method

- 2.1. The 237 Leaf Wetness Sensor design emulates the surface area of a leaf. The dimensions of the sensor are 2.75" W x 3" L x 0.25" D, and its weight is 0.5 lb. Surface wetness is detected using a resistance grid (see Fig. 1 at right) of interlaced gold-plated copper etched ‘fingers’ on an electronic circuit board. Condensation connects the etched ‘fingers’ of the board, resulting in a resistance change. The change in resistance is detected by change in voltage of a half bridge of fixed resistors (built into sensor cable) excited by AC current.

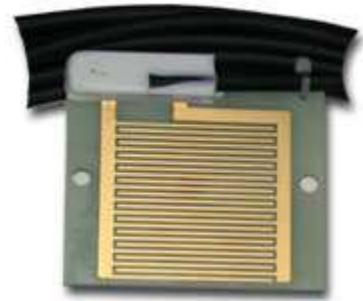


Figure 1.
Leaf Wetness Sensor

3. Definitions

- | | | |
|------|-------|---|
| 3.1. | CS | Campbell Scientific, Inc |
| 3.2. | NEMA | National Electrical Manufacturers Association |
| 3.3. | PAAQL | Purdue Agricultural Air Quality Laboratory |
| 3.4. | PAML | Purdue Applied Meteorology Laboratory |
| 3.5. | QAPP | Quality Assurance Project Plan |
| 3.6. | SOP | Standard operating procedure |

4. Cautions

- 4.1. Be careful when connecting the sensor cable to the data logger, being sure the connections are tight and made according to SOP W6.
- 4.2. Replace the sensor if the cable should become detached from the sensor (do not attempt to resolder it to the sensor), as the cable contains built-in components (a half bridge of fixed resistors) specifically designed to make the sensor operate correctly.
- 4.3. As with any electrical device, use proper care when crimping cables and connectors.

5. Interferences

- 5.1. Dust will reduce the sensitivity of the sensor to drying conditions.
- 5.2. Fingerprints and grease will reduce the sensitivity of the sensor to wetting conditions.

6. Personnel Qualifications

- 6.1. Each member of the field staff must read the sensor instruction manual and this SOP before operating this equipment.
- 6.2. Personnel must be trained in the use of the handheld multimeter before initiating the procedure. Training requires 10 min.

7. Equipment and Supplies

- 7.1. Leaf Wetness Sensor (Campbell Scientific, Model 237, Logan, Utah)
- 7.2. #2 common head screwdriver
- 7.3. 1/8" slotted screwdriver
- 7.4. Bottle of distilled water for cleaning and testing sensor
- 7.5. Multimeter

8. Procedures

- 8.1. Preparation for use
 - 8.1.1. Clean the Model 237 Leaf wetness sensor thoroughly using acetone; allow to dry.
 - 8.1.2. Coat the sensor with a single coat of standard Latex exterior house paint; allow to thoroughly dry.
- 8.2. Acceptance
 - 8.2.1. Attach multimeter probes to red and black wires of sensor cable.
 - 8.2.2. Set multimeter to measure resistance.
 - 8.2.2.1. Read sensor response on multimeter when sensor is dry.
 - 8.2.2.1.1. Value should be greater than 3,100,000 Ω .
 - 8.2.2.1.2. If less than 3,100,000 Ω , allow sensor to dry in a warm environment for a minimum of 24 h. If the sensor still reads less than 3,100,000 Ω , return sensor to manufacturer.
 - 8.2.2.2. Wet sensor with tap water and read sensor response on multimeter.
 - 8.2.2.2.1. Value should be in the range of 120,000 – 500,000 Ω ($\pm 40,000$ Ω).
 - 8.2.2.2.2. If wet resistance is $< 80,000$ Ω or $> 540,000$ Ω , return sensor to manufacturer.
- 8.3. Calibration check
 - 8.3.1. Note that this device is not a precision instrument, and exact resistance readings may vary considerably, depending on a wide variety of conditions including the mineral content of the distilled test or rain water.
 - 8.3.2. Attach red and black sensor wires to probe wires of multimeter.
 - 8.3.3. Set multimeter to measure resistance.
 - 8.3.3.1. Read sensor response on multimeter when sensor is dry.

- 8.3.3.1.1. Record value on calibration check sheet (Appendix A). If resistance is over-range on the multimeter's highest resistance scale, record resistance as infinite.
- 8.3.3.1.2. Value should be greater than 3,100,000 Ω .
- 8.3.3.1.3. If not, return sensor to manufacturer.
- 8.3.3.2. Wet sensor with distilled water and read sensor response on multimeter.
 - 8.3.3.2.1. Record value on calibration check sheet (Appendix A)
 - 8.3.3.2.2. Value should be within 120,000 Ω – 500,000 Ω (wet resistance tolerance is not critical).
 - 8.3.3.2.3. If after one minute, resistance value is less than 80,000 Ω , or over 1,000,000 Ω , return sensor to manufacturer.
- 8.4. Installation
 - 8.4.1. Mount sensor on the 3-m (10-ft) meteorological mast (SOP U5) at 2-m (6.6-ft) height, according to SOP W6.
 - 8.4.2. Pass cable along ground to weatherproofed NEMA 4X box (SOP W6) and through specified knockout.
 - 8.4.3. Attach sensor wires to data logger according to SOP W6 using the 1/8" slotted screwdriver.
 - 8.4.3.1. Connect black wire to 5 VAC excitation, purple wire to ground, clear wire to ground, and red wire to analog single ended measurement channel.
 - 8.4.3.2. Data logger will log voltage returned from the wetness sensor's output wire (red wire) as a ratio of the measured voltage divided by the excitation voltage (V_s/V_x). The datalogger applies 5 VAC to the sensor's exciter (black) wire.
 - 8.4.3.2.1. Sensor resistance (R_s) is recorded as a voltage ratio using the following calculation: $V_s/V_x = R_2/(R_2+R_1+R_s)$.
 - 8.4.3.2.2. R_1 and R_2 are fixed values; $R_1=100,000 \Omega$ and $R_2=1,000 \Omega$ (Fig. 1 of Model 237 manual).
 - 8.4.3.2.3. For example, if the sensor resistance is 101,200 Ω , the voltage ratio recorded would be 0.0049455 ($1000(R_2)/(102,200(R_2+R_1+R_s))$).
 - 8.4.3.3. This logged voltage will indicate the condition the sensor is currently detecting.
 - 8.4.3.4. Values from 4.524 mV to 1.663 mV ($120,000 \Omega < R_s < 500,000 \Omega$) will indicate "Wet" conditions.
 - 8.4.3.5. Values less than 0.3124 mV ($R_s > 3,100,000 \Omega$) will indicate "Dry" conditions.
 - 8.4.3.6. Values between 1.663 mV and 0.3124 mV ($500,000 \Omega < R_s < 3,099,999 \Omega$) will indicate transition "Conditions other than wet or dry".
 - 8.4.4. While sensor is dry, check to see that data logger is indicating "Dry" conditions. Check wiring connections for error if response is incorrect.
 - 8.4.5. Rinse the wetness grid with tap water and verify data logger's proper "Wet" response. Check wiring connections for error if response is incorrect.
- 8.5. Troubleshooting
 - 8.5.1. If readings are noisy or erratic, check for poor connections.
 - 8.5.2. If sensor does not respond to wetness, check for broken wires or connections.
- 8.6. Data acquisition, calculations & data reduction requirements
 - 8.6.1. Analog input is monitored by the datalogger.

- 8.6.1.1. Ratio of excitation to sensor resistance is recorded and then calculated at PAML to be “Wet”, “Dry” or “Conditions other than wet or dry”, based on sensor resistance calculated from recorded voltage ratio.
- 8.6.2. Datalogger will record measurements every 5 minutes.
- 8.7. Computer hardware & software.
 - 8.7.1. Attach the sensor to a Campbell Scientific datalogger through electrical wires that will extract data from the instruments.
 - 8.7.2. The Campbell Scientific data logger will record the data and store it.
 - 8.7.3. Datalogger operates using on-board BASIC-like programming.
 - 8.7.3.1. The general program for measurements is stated in the Model 237 Manual.

9. Data and Records Management

- 9.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method, and in a record book designated for meteorological sensors.
- 9.2. Manage all data according to SOP D1.
- 9.3. Document all data and information on field notes with permanent ink, or in electronic notes. Overstrike errors in ink with a single line, then initial and date the overstrike.

10. Quality Control and Quality Assurance

- 10.1. Daily check from PAML
 - 10.1.1. If value is -6999, flag with “1”.
 - 10.1.1.1. Voltages over the maximum measurable input are indicated by data logger as -6999, and indicate sensor failure (short circuit). Dispatch personnel to check sensor and sensor wiring on site.
 - 10.1.2. Check weather data from an outside source for the area such as <http://www.crh.noaa.gov/iwx/index.php?mystation=KLAF> (Lafayette, IN). If indicated data does not match conditions shown by outside weather sources on two or more days:
 - 10.1.2.1. Flag recorded value with a “2” as invalid.
 - 10.1.2.2. Dispatch personnel to check sensor and sensor wiring on site.
 - 10.1.2.2.1. If damage is indicated, repair or replace as indicated in Section 8.4.
- 10.2. Perform the following checks at the beginning and end of each measurement period.
 - 10.2.1. Check to be sure probe surface is clean.
 - 10.2.1.1. If dirty, clean with distilled water and cloth.
 - 10.2.2. Check integrity of the plastic casing of the signal cable.
 - 10.2.2.1. If damaged slightly, repair with heat-shrink tubing.
 - 10.2.2.2. If damaged significantly, replace sensor and return sensor to manufacturer for repair.
 - 10.2.3. Wet sensor with tap water.
 - 10.2.3.1. Check datalogger for value corresponding to a wet sensor
 - 10.2.3.2. If data logger does not indicate wet conditions, check wiring (Section 8.2).
 - 10.2.4. Dry sensor.
 - 10.2.4.1. Check datalogger for value corresponding to a dry sensor.

- 10.2.4.2. If data logger does not indicate dry conditions, check wiring (Section 8.2).
- 10.2.5. Record results of checks on the site logbook and electronic spreadsheet.

11. References

- 11.1. Campbell Scientific. 1996. Model 237 Leaf Wetness Sensor Instruction Manual. Rev. 2/2006. Available online at www.campbellsci.com/documents/manuals/237-lc.pdf . Accessed on 2/23/2006.
- 11.2. SOP D1. 2006. Management of Weather and Lagoon-Characterization Data. Standard Operating Procedure D3. Purdue Applied Meteorology Lab.
- 11.3. SOP U5. 2006. The Installation of Open-source Measurement Equipment. Standard Operating Procedure U5. Purdue Applied Meteorology Lab.
- 11.4. SOP W6. 2006. Establishment, Data Acquisition and Control of Weather and Lagoon Characterization Hardware. Standard Operating Procedure W6. Purdue Applied Meteorology Lab.

12. Contact Information

- 12.1. Distributor: Campbell Scientific, Inc., 815 West 1800 North, Logan, Utah, 84321
 - 12.1.1. Technical support: 435-753-2342

**THE MEASUREMENT OF BAROMETRIC PRESSURE WITH THE SETRA
MODEL 278 (CAMPBELL SCIENTIFIC CS100) BAROMETER**

Standard Operating Procedure (SOP) W5

**THE MEASUREMENT OF BAROMETRIC PRESSURE
WITH THE SETRA MODEL 278 (CAMPBELL SCIENTIFIC CS100) BAROMETER
Standard Operating Procedure (SOP) W5**

**Prepared by
Richard H. Grant**

**Reviewed by
Albert. J. Heber and Bill W. Bogan**

Effective Date: November 6, 2006

**PURDUE APPLIED METEOROLOGY LABORATORY (PAML)
Agronomy, Purdue University
West Lafayette, IN 47907**

Table of Contents

	Page
1. Scope and Applicability.....	3
2. Summary of Method.....	3
3. Definitions.....	3
4. Cautions.....	4
5. Interferences.....	4
6. Personnel Qualifications.....	4
7. Equipment and Supplies.....	4
8. Procedures.....	5
9. Data and Records Management.....	7
10. Quality Control and Quality Assurance.....	7
11. References.....	8
12. Contact Information.....	8
Appendix A. Fortin Barometer Operation.....	9
Appendix B. Calibration Record: Setra 278 Barometric Pressure Sensor.....	12

1. Scope and Applicability

- 1.1. Barometric pressure measurements are needed to determine the mass concentration of gases from volume concentration measurements.
- 1.2. The resonant frequency of silicon is a useful means of measuring pressure.
- 1.3. The lower detection limit for the instrumentation described in this SOP is 600 hPa.
- 1.4. The range of the instrument, under normal atmospheric conditions, is from 600 to 1100 hPa.
- 1.5. Sensor response is 5mV / hPa.
- 1.6. Sensor accuracy is ± 3 hPa
- 1.7. This procedure applies only to the Setra model 278 barometric pressure sensor, purchased as CS100 Barometric Pressure Sensor
- 1.8. This procedure applies to the open source portion of the National Air Emissions Study.

2. Summary of Method

Barometric pressure is measured using two closely-spaced, parallel, electrically-isolated metallic surfaces, one of which is essentially a diaphragm capable of slight flexing under applied pressure. The surfaces are mounted to allow slight mechanical flexing by small changes in pressure. Changes in the gap between the surfaces change the electrical capacitance of the gap, which is detected and converted to a proportional high level analog signal. The Setra model 278 sensor is used under this SOP because it is well-suited to long-term environmental applications, as its housing and all of its wettable parts are constructed of stainless steel or polyester. The sensor itself is enclosed in a thermally stable, low-hysteresis glass fused ceramic capsule which minimizes temperature effects on pressure readings.

3. Definitions

ARM	Atmospheric Radiation Monitoring Network
CS	Campbell Scientific, Inc
hPa	The S.I. unit of pressure is Pascal. The worldwide meteorological community uses the hectopascal(hPa) i.e. a hundred Pascals, which is the metric equivalent of a millibar. "Standard atmospheric pressure=1013 hPa = 101,325 Pa= 101.3 kPa=760 mm Hg=14.696 psi."
NEMA	National Electrical Manufacturers Association
PAAQL	Purdue Agricultural Air Quality Laboratory
PAML	Purdue Applied Meteorology Laboratory
QAPP	Quality Assurance Project Plan
VDC	Direct Current voltage

4. Cautions

- 4.1. Static charge damages the sensor. To avoid damage, take adequate anti-static measures when handling.
 - 4.1.1. Wear an antistatic wrist strap that is properly connected to earth ground when in contact with the sensor.
 - 4.1.2. If an antistatic wrist strap is not available, discharge static electricity by touching a grounded metal object before touching the sensor itself.

5. Interferences

- 5.1. Failure to provide an open vent at the bottom of the NEMA 4X box will interfere with barometric measurements by blocking the sensor's ability to sense atmospheric pressure changes.
- 5.2. Failure to protect sensor from condensation or liquid water may cause permanent damage. See Section 8.2.2 for proper installation to avoid condensation. Place desiccant inside the NEMA box to absorb any moisture which does occur.

6. Personnel Qualifications

- 6.1. Personnel must be trained in the use of the handheld frequency multimeter before initiating the procedure. Training requires 10 min.
- 6.2. Personnel must be trained in the use of the power supply before initiating the procedure. Training requires 10 min.
- 6.3. Each staff member must read and understand the sensor instruction manual and this SOP before operating this sensor.

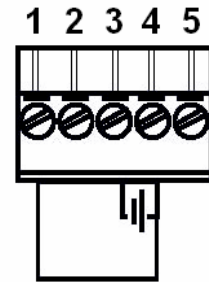
7. Equipment and Supplies

- 7.1. Multimeter
- 7.2. #2 Phillips head screwdriver
- 7.3. 1/8" slotted screwdriver
- 7.4. 2-inch length of jumper wire
- 7.5. 12 VDC power supply
- 7.6. 2-in. section of plastic tubing
- 7.7. Toothbrush
- 7.8. Electrical cleaner
- 7.9. Fortin Barometer (kept at PAML)
- 7.10. CS100 Barometric Pressure Sensor
- 7.11. NEMA 4X Box
- 7.12. Campbell Scientific Data Logger

8. Procedures

8.1. Acceptance

- 8.1.1. Connect terminal 4 to positive terminal of 12 VDC supply and terminal 3 to power supply ground (See diagram at right).
- 8.1.2. Connect a jumper across terminals 1 and 4.
- 8.1.3. With multimeter off, connect terminal 2 to negative terminal of multimeter and terminal 5 to positive terminal of multimeter.
- 8.1.4. Turn multimeter on and set to mV DC.
- 8.1.5. After 2 s, read measured voltage on multimeter.
- 8.1.6. Voltage should be approximately 1-2.25 VDC (800-1050 hPa).
 - 8.1.6.1. If values are less than 1V or greater than 2.25 VDC, the sensor is defective, and should be returned to the manufacturer for replacement or repair.



8.2. Setup

- 8.2.1. Mount the instrument on the inside of the NEMA 4X box on the 10-foot mast according to SOP U5.
- 8.2.2. Mount the sensor with the pneumatic connector pointing vertically downwards to prevent condensation collecting on the pressure cavity and ensure that water cannot enter the sensor. Place fork lug under the mounting screw for grounding.
- 8.2.3. Using the 1/8" slotted screwdriver, connect the red wire to 12 VDC supply, black to power ground, yellow to signal ground, clear to shield, green to the external trigger (control terminal), and blue to a single-ended analog terminal of the data logger, according to SOP W6.
- 8.2.4. On power-up, the sensor requires less than 1 second of warm-up time.
- 8.2.5. The voltage across the blue and yellow wires (when terminals 1 and 4 are jumper wired as in Step 8.1.2) should be approximately 1-2.25 VDC (800-1050 hPa).
 - 8.2.5.1. If values are less than 1 VDC, the sensor is defective, and should be returned to the manufacturer for replacement or repair.

8.3. Calibration

- 8.3.1. Calibrate the sensor every 6 mos., or at a different frequency if specified by the QAPP for a particular project.
- 8.3.2. Calibrate the sensor system (sensor and data logger) against a Fortin Barometer at PAML.
 - 8.3.2.1. Follow the procedures for reading and adjusting the Fortin barometer documented in Appendix A of this SOP.
 - 8.3.2.2. Read instantaneous measurements from datalogger/ sensor system at the time of each Fortin Barometer reading.
- 8.3.3. Conduct six paired measurements over a two-day period, average mean of their respective readings are taken out and if this matches then it is calibrated.
- 8.3.4. Record the results of the calibration on the sensor lab notebook form (Appendix B).
 - 8.3.4.1. If the mean difference between Fortin barometer and sensor/datalogger measurement is less than or equal to 2 hPa, then the sensor passes calibration and the drift is back-corrected according to Equation 1.

$$P_{corr}(t) = P(t) \left[1 - \frac{P_{measured}(i) - P_{expected}(i)}{P_{expected}} \right] \left(\frac{t}{p} \right) \quad (\text{Equation 1})$$

Where:

- P_{corr} = Back-corrected value at time t into the last measurement period
 $P(t)$ = Original measured value at time t
 $P_{measured}$ = Measured barometric pressure during calibration comparison
 $P_{expected}$ = Barometric pressure indicated by the Fortin barometer during calibration comparison
 p = Length of time between calibrations

8.3.4.2. If the mean difference between Fortin barometer and sensor/datalogger measurement is >2 hPa, return sensor to manufacturer for repair or replacement.

8.3.4.2.1. If the mean difference is <6 hPa, adjust measurements since the last passed calibration linearly over time to the current calibration to account for the drift according to Equation 1. Correction coefficients determined at this point can be entered into the PAML computers to correct data collected during the next measurement period; the QAPP for a particular project will specify whether or not this is to be done.

8.3.4.2.2. If the mean difference is >6 hPa, invalidate barometric pressure measurements over the preceding measurement period.

8.4. Troubleshooting

8.4.1. If pressure measurements vary widely over 24 h, check that the NEMA box is not 'hermetically sealed'. A hermetic seal is evident when air is distinctly forced out at the sides of the opening when the box is closed.

8.4.2. Be sure there is no condensate in the NEMA box.

8.4.3. Visually inspect the casing for damage.

8.4.4. Ensure there is a secure, clean ground connection.

8.4.5. Ensure that the pneumatic connection and tubing are secure and undamaged.

8.4.6. Replace desiccant in the NEMA box as needed.

8.5. Data acquisition, calculations & data reduction requirements

8.5.1. Monitor the analog voltage output with a Campbell Scientific datalogger or equivalent data acquisition system (See SOP W6).

8.5.2. Take measurements every 5 min

8.5.3. Report voltage data to five significant digits.

8.6. Computer hardware & software

8.6.1. Attach the sensors through electrical wires to a Campbell Scientific datalogger (or equivalent data acquisition system) that will extract data from the instruments.

8.6.2. The logger program for reading the sensor is written in a version of BASIC language.

8.6.2.1. The program converts the sensor voltage to hPa using the following formula:

$$\text{hPa} = 600 + 0.2 \text{ mVDC} \quad (\text{Equation 2})$$

- 8.6.2.2. The sensor units are logged as hPa.
- 8.6.3. Record and store the data with the datalogger.

9. Data and Records Management

- 9.1. Maintain all laboratory records in a worksheet of the electronic field notes spreadsheet designated for this method, as well as in a bound record book designated for meteorological sensors.
- 9.2. Manage data according to SOP D1.
- 9.3. Record the calibration information on the form provided in Appendix B.
 - 9.3.1. Maintain a separate record sheet for each individual sensor.
- 9.4. Document all data and information on field data sheets and in site logbooks with permanent ink, or in electronic notes.
- 9.5. Overstrike errors in writing with a single line. Initial and date all corrections.

10. Quality Control and Quality Assurance

- 10.1. Daily
 - 10.1.1. If value is missing, flag with “1”.
 - 10.1.2. If value is less than 600 hPa or greater than 1100 hPa (i.e. outside the range of the sensor), then flag value with “2”.
 - 10.1.3. Calculate the altitude-adjusted barometric pressure from the nearest National Weather Service Weather Station.
 - 10.1.3.1. Correct NWS measured pressure to height of measurement location according to Equation 2 (Holton, 1979):

$$P_{site} = P_{NWS} e^{-(Z_{site} - Z_{NWS})/H} \quad \text{(Equation 3)}$$
 Where:
 - P_{site} = Height-adjusted NWS pressure for site
 - P_{NWS} = Pressure at NWS station
 - Z_{site} = Site elevation
 - Z_{NWS} = NWS station elevation
 - H = $287(T_{NWS})/9.80665$
 - T_{NWS} = Absolute temperature (°K) at NWS station.
 - 10.1.4. If the altitude-adjusted difference between the barometric pressure at the sampling site and the nearest local weather station is > 4 hPa, then flag the day’s data with “3”.
 - 10.1.4.1. Further investigation of the cause for the difference (such as synoptic weather front over the area) will be conducted.
- 10.2. Repeat the Acceptance Check (Section 8.1) before beginning a measurement period (i.e. during the setup of equipment at a site).
- 10.3. Do the following at the beginning and end of each measurement period:
 - 10.3.1. Visually inspect the casing for damage.
 - 10.3.1.1. Replace sensor if damaged.

- 10.3.2. Ensure there is a good secure and clean ground connection.
 - 10.3.2.1. Secure ground if needed.
- 10.3.3. Ensure that the pneumatic connection and tubing is secure and undamaged.
 - 10.3.3.1. Replace tubing if needed.
- 10.3.4. Check dessicant in the NEMA box.
 - 10.3.4.1. Replace as needed.
- 10.3.5. Check cleanliness of connectors.
 - 10.3.5.1. If corroded, clean with toothbrush and electrical cleaner.

11. References

- 11.1. ARM. 2005.: Temperature, Humidity, Wind, and Pressure System Handbook. Atmospheric Radiation Measurement Climate Research Facility, ARM TR-030, 13p.
- 11.2. Campbell Scientific. 2005. Model CS100 Barometric Pressure Sensor Instruction Manual, 8p.
- 11.3. Holton, J.R. 1979. An introduction to dynamic meteorology, 2nd Ed., Academic Press,
- 11.4. Setra Systems Inc. 2004. Installation guide: Model 278 Barometric Pressure Sensor, 5p.
- 11.5. National Physical Laboratory. 2006. "Frequently asked questions - pressure and vacuum: How do I use a Fortin or Kew Pattern mercury barometer?", <http://www.npl.co.uk/pressure/faqs/usehgbaro.html#calcpress>, accessed 1/11/2006
- 11.6. SOP U5. 2006. The Installation of Open-source Measurement Equipment. Standard Operating Procedure U6. Purdue Applied Meteorology Lab.
- 11.7. SOP W6. 2006. Establishment, Data Acquisition and Control of Weather and Lagoon Characterization Hardware. Standard Operating Procedure W6. Purdue Applied Meteorology Lab.
- 11.8. SOP D1. 2006. Management of Open-Source, Weather, And Lagoon-Characterization Data. Standard Operating Procedure D1. Purdue Applied Meteorology Lab.
- 11.9. USDOC Weather Bureau. 1963. Manual of Barometry (WBAN) Volume 1, 1st edition, 1963
- 11.10. USEPA. 1989. Quality Assurance Handbook For Air Pollution Measurement Systems, Volume IV- Meteorological Measurements.
- 11.11. USEPA. 2000. Meteorological Monitoring Guidance for Regulatory Modeling Applications, EPA-454/R-99-005.

12. Contact Information

- 12.1. Distributor: Campbell Scientific, Inc., 815 West 1800 North, Logan, UT, 84321
 - 12.1.1. Technical support: 435-753-2342
- 12.2. Manufacturer: Setra Systems, Inc., 159 Swanson Road, Boxborough, MA 01719
 - 12.2.1. Repair Dept. Fax: 978-266-2158/Phone: 978-266-2194

Appendix A. Fortin Barometer Operation

The correct procedure for measuring pressure with a Fortin Barometer is:

- Adjust the cistern mercury surface to within a small distance of the fiducial point.
- Tap the barometer (to achieve a stable meniscus).
- Set the fiducial point.
- Set the vernier.
- Measure the barometer's temperature (and add calibration correction).
- Read the vernier.
- Apply the calibration correction for the barometer.
- Apply the temperature and gravity corrections (from tables or formula).

Fortin barometers should be used with their axis of rotation vertical - such that a setting of the mercury surface to the fiducial point remains correct after axial rotation through any angle.

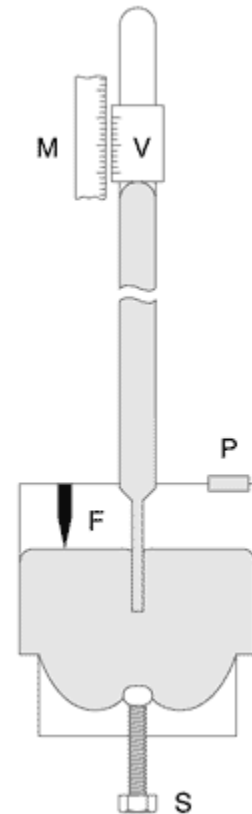
Setting the Fiducial Point (F in diagram at right)

The lower mercury surface in a Fortin barometer has to be set to a datum level before adjusting its vernier (see schematic diagram) and the accuracy of pressure measurement depends crucially upon proper setting.

To achieve this, the mercury surface should first be lowered until it is clearly *below* the fiducial point (F in diagram at right). After tapping the barometer, the level-adjusting screw (S in diagram at right) should be turned slowly until the thin background of light between the mercury surface and the point just disappears, while viewed along a horizontal line of sight. To confirm proper setting, observe the point of contact at an angle of elevation of about 30° and determine whether or not the fiducial point is making an indentation in the mercury.

When the setting is correct there should be no more than the slightest dimple where the fiducial point makes contact with the mercury surface. If the surface is bright and the setting correct, the tip of the point will appear to coincide with its reflected image in the mercury surface. Correct setting is achieved when the mercury surface is *raised* to the fiducial point but not lowered - if the mercury surface overshoots the point it should be lowered and then raised again.

Setting the vernier: With suitably bright background illumination (using flashlight aimed at the white plastic background for the vernier), and taking care to correctly align the front and back setting edges, the vernier cursor (V in diagram at right) should be lowered until it appears on the



summit of the mercury meniscus. Proper setting can be aided by making small 'pecking' head movements to ensure that both cursor edges just meet the meniscus. Read the vernier.

Measuring temperature: Mercury barometers have a temperature coefficient of nearly 0.2 hPa per °C so it is important to ensure that their thermometers respond to ambient temperature changes in the same fashion as the barometer. Read the temperature off the thermometer attached to the barometer case.

Calculation of pressure from barometer readings: Three corrections to the measured barometric pressure must be made:

- Temperature correction
- Calibration correction
- Gravity correction

Theoretically, the corrections are made according to the following equations:

$$t = (t' - 32) * 5/9$$

$$\text{Pressure} = \frac{g}{9.80665} \left\{ R + c - R \frac{(b - a)t}{(1 + bt)} \right\}$$

where R is the barometer reading (in inches Hg)

c is its calibration correction (+.003 in Hg for barometer at PAML)

g is the acceleration due to gravity at the level of the barometer in m/s^2 (see below)

b is the coefficient of expansion of mercury (taken to be $0.0001818 \text{ } ^\circ\text{C}^{-1}$ for meteorological purposes)

a is the coefficient of linear expansion of the scale (taken to be $0.0000184 \text{ } ^\circ\text{C}^{-1}$ for brass)

t is the temperature of the barometer in °C

t' is the temperature of the barometer in °F

$$\text{and } g = 9.7803184 (1 + A \sin^2 L - B \sin^2 2L) - 3.086 \times 10^{-6} H$$

where $A = 0.0053024$

$B = 0.0000059$

$L = \text{Latitude (40.5)}$

$H = \text{height of barometer meters above sea level (197 m)}$

At West Lafayette, $g = 9.80153$, so the gravity correction is $9.80153 / 9.80665 = 0.999$.

The correction terms involving a, b, and c were combined in the following table. To correct for the barometer local gravity and temperature, multiply the measured value by the value from the following tables and subtract from the measured value, then multiply by 0.999.

deg. F	correction	deg. F	correction	deg. F	correction
50	0.001631	70	0.003436	90	0.005234
50.5	0.001676	70.5	0.003481	90.5	0.005279
51	0.001721	71	0.003526	91	0.005324
51.5	0.001767	71.5	0.003571	91.5	0.005369
52	0.001812	72	0.003617	92	0.005414
52.5	0.001857	72.5	0.003662	92.5	0.005459
53	0.001902	73	0.003707	93	0.005504
53.5	0.001947	73.5	0.003752	93.5	0.005548
54	0.001993	74	0.003797	94	0.005593
54.5	0.002038	74.5	0.003842	94.5	0.005638
55	0.002083	75	0.003887	95	0.005683
55.5	0.002128	75.5	0.003932	95.5	0.005728
56	0.002173	76	0.003977	96	0.005772
56.5	0.002219	76.5	0.004022	96.5	0.005817
57	0.002264	77	0.004067	97	0.005862
57.5	0.002309	77.5	0.004111	97.5	0.005907
58	0.002354	78	0.004156	98	0.005952
58.5	0.002399	78.5	0.004201		
59	0.002444	79	0.004246		
59.5	0.002489	79.5	0.004291		
60	0.002535	80	0.004336		
60.5	0.00258	80.5	0.004381		
61	0.002625	81	0.004426		
61.5	0.00267	81.5	0.004471		
62	0.002715	82	0.004516		
62.5	0.00276	82.5	0.004561		
63	0.002805	83	0.004606		
63.5	0.00285	83.5	0.004651		
64	0.002896	84	0.004696		
64.5	0.002941	84.5	0.004741		
65	0.002986	85	0.004786		
65.5	0.003031	85.5	0.004831		
66	0.003076	86	0.004875		
66.5	0.003121	86.5	0.00492		
67	0.003166	87	0.004965		
67.5	0.003211	87.5	0.00501		
68	0.003256	88	0.005055		
68.5	0.003301	88.5	0.0051		
69	0.003346	89	0.005145		
69.5	0.003391	89.5	0.00519		

